REMOVAL OF RESIDUAL TEXTILE DYE EFFLUENT USING ACTIVATED CARBON PREPARED FROM AGRICULTURAL RESIDUES

A Thesis

Submitted to the

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In partial fulfillment of the requirements for the degree of

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IN

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By

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CERTIFICATE - I

This is to certify that the thesis entitled **"Removal of residual textile dye effluent using activated carbon prepared from agricultural residues"** submitted to the College of Community Science, Assam Agricultural University in partial fulfillment for the degree of **Doctor of Philosophy (Community Science)** in **Textiles and Apparel Designing,** is a record of research work carried out by **Reena Roy** under my personal supervision and guidance.

All helps received by her have been duly acknowledged.

No part of this thesis has been reproduced elsewhere for any degree.

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ABSTRACT

Textile dyeing industries are one of the most polluted industries which createlots of problems for environmental pollution. The textile dyeing industry produces in large amount of production and release of waste water effluent. In the present study, activated carbons were prepared from almond, coconut, mustard, rice bran and sesame oil cakes. The powdered oil cakes were treated with 5% NaCl for 12 hours in the rotary shaker followed by chemical activation with 1 N H_3PO_4, 2.5 N H_2SO_4 and 2.5 N H_3PO_4 for 24 hours. The pyrolysis was performed at 300˚C for 2 hours. The ball milling technique was applied to reduce the particle size of the activated carbon. The integrated activated carbons were used for the color removal for acid and metal complex dye effluent from the wool dyeing unit. For dye effluent treatment different concentrations of adsorbent *viz.*,0.1%, 0.25%, 0.5% and 1.0% and time period 30, 60 and 90 minutes were taken respectively. The results showed that very small quantities (1.0 and 2.0%) of activated carbons were sufficient to remove around 92% color from the dye effluent. The particle size of the activated carbon was further reduced by ball milling in Pulverisette 6 for 1 hour.The characterization of activated carbon was synthesized such as FTIR, SEM, EDX, XRD, BET, bulk density, porosity, ash content, moisture content, pH, zero point charge (pzc), iodine number, methylene blue, particle size analysis, COD and BOD respectively. The activated carbon characterized by using scanning electron microscopy (SEM) revealed the small pore size with higher surface area that indicates lower absorbency. FTIR analysis also revealed the presence of various types of functional groups during different activation temperatures. From EDX analysis, a negligible quantity of Na, K and S in 2.5 N H_3PO_4 almond activated carbons and in 2.5 N H_2SO_4 almond activated carbons there was a negligible quantity of Mg, Si, P and C and other elements were present.The X-ray diffraction pattern of the 2.5 N H_2SO_4 and2.5 N H_3PO_4 almondwere recorded at peak 25˚ and 29˚ respectively, and indicated the presence of amorphous structure of the activated carbon with the diffraction pattern of (002).In BET analysis; the surface area, pore radius and pore volume of 2.5 N H_3PO_4 was recorded as 16.14m^2/g, 0.85nm and 0.029cc/g and in 2.5 N H_2SO_4 almond, it was

recorded 64.28m^2/g, 0.72nm and 0.040cc/g respectively. Bulk density 4.533g/cm^3, porosity 4.500%, ash content 0.176%, moisture contents 0.040%, methylene blue 123.667mg/g, pH 6.180, zero point charge 4.140pzc, particle size 310.333nm, iodine number 22.067m^2/g of 2.5 N H_3PO_4 almond (ball milling) activated carbon were recorded best compared to 1 N H_3PO_4 coconut (ball milling) and 2.5 N H_3PO_4 mustard (ball milling) activated carbon. The bulk density 5.233g/cm, porosity 3.633%, ash content 0.172%, moisture contents 0.071%, methylene blue 116.000mg/g, pH 6.973, zero point charge 6.460pzc, particle size 825.000nm, and iodine number 21.5333m^2/g were also recorded best in 2.5 N H_2SO_4 almond (ball milling) activated carbon metal complex dye effluent compared to 2.5 N H_3PO_4 rice bran (ball milling) and 2.5 N H_2SO_4 sesame (ball milling) activated carbon. The good absorbency was recorded 0.107 at 2.0% concentration and 90 minutesof contact time in 2.5 N H_3PO_4 almond (ball milling) activated carbon acid dye effluent compared to 1 N H_3PO_4 coconut (ball milling) and 2.5 N H_3PO_4 mustard (ball milling) activated carbon. The pH and TDS of the effluent were recorded 4.523 and 2668.66mg/L, concentration 2.0% in 60 minutes, which were found to be best among the 1 N H_3PO_4 coconut (ball milling) and 2.5 N H_3PO_4 mustard (ball milling) activated carbon in acid dye effluent. The good absorbency was recorded 0.153 concentration 1.0% and 90 minutes of contact time in 2.5 N H_2SO_4 almond (ball milling) activated carbon metal complex dye effluent compared to 2.5 N H_3PO_4 rice bran (ball milling) and 2.5 N H_2SO_4sesame (ball milling) activated carbon. The pH and TDS of the effluent were recorded best in5.233 and 2219.33mg/L, concentration 1.0% at 90 and 30 minutes respectively in 2.5 N H_2SO_4 almond (ball milling) activated carbon metal complex dye effluent. Therefore, 2.5 N H_3PO_4 almond (ball milling) activated carbon acid dye and 2.5 N H_2SO_4 almond (ball milling) activated carbon metal complex dye effluent proved to be an excellent oil cake for preparation of activated carbon in the textile dye industry and can be used as an alternative source for wastewater treatment.

Keywords: Activated carbon, oil cake, wastewater effluent, FTIR, SEM, XRD, TDS

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NOMENCLATURE

FTIR	-	Fourier Transform Infrared Spectroscopy
BET	-	BrunauerEmmet Teller
pH_{PZC}	-	pH of the point of zero charge
SEM	-	Scanning electron microscope
EDX	-	Energy Dispersive X-Ray Analysis
XRD	-	X-Ray Diffraction
H_2SO_4	-	Sulfuric acid
H_3PO_4	-	Phosphoric acid
NaCl	-	Sodium Chloride
TDS	-	Total Dissolve Solids
COD	-	Chemical Oxygen Demand
BOD	-	Biological Oxygen Demand
C	-	Concentration
T	-	Time
TDS	-	Total Dissolved Solids
HNO3	-	Nitric acid
Nylon mesh	-	*Mesh* count of the *Nylon* Micron Filter *Mesh* refers to how many holes in a linear inch
Heating mantal	-	A *heating mantle*, or isomantle, is a piece of laboratory equipment used to apply heat to containers, as an alternative to other forms of heated bath.
Char	-	To convert to charcoal or carbon usually by heat and burn.

CHAPTER - I

INTRODUCTION

Textile dyeing industries are one of the most polluted industries which create lots of problems for environmental pollution. The textile dyeing industry produces in large amount of production and release of waste water effluent. Wastewater is generally released directly into the nearby drains, rivers, stagnant ponds, or lagoons. Such wastewater disposal may cause damage to the quality of the receiving water bodies, the aquatic eco-system, and the biodiversity of the environment. The adsorption process using various agricultural waste products can be applied for the removal of dye effluent from wastewater.

Before disposal of wastewater to the water bodies or land, the textile industrial effluent went through a primary, secondary, and tertiary treatments. Activated carbons are widely used as an adsorbent for the removal of color from dye effluent through separation and purification of gas and liquid which are having potential applications in the textile industry. Biological, physical, and chemical methods are also used for textile wastewater treatment which includes microbial degradation, membrane filtration, oxidation, and ozonation. Conventional treatments are frequently unfavorable for removal of color from dyes due to their high solubility and low biodegradability.

Dyeing industry effluents constitute one of the most difficult tasks for wastewaters to be treated not only for their high chemical oxygen demand (COD) and biological oxygen demands (BOD), suspended solids content, and toxic compounds but also for color. The high cost of importing water treatment reduces chemical consistently and increases the quality of drinking water. It may cause acute and chronic effects on human skin such as allergies, dermatitis, skin irritation, cancer, mutation, etc. The textile industry is the most polluting and chemically intensive industrial sector (Uzal, 2015).

The textile industry for wet processing operations consumes huge volumes of water and chemicals. Wastewater effluent coming from different sources contained

chemicals such as acids, alkalis, colors, high BOD/COD concentration, surfactants, dispersing agents, soap and higher amount of metals (Paul *et al.*, 2012). Thus, recycling of treated wastewater must be done due to the high levels of contamination in the dyeing and finishing process. The presence of dye levels at a very low concentration is highly observable and undesirable.

Color is the first toxin to be removed from the wastewater before it is discharged into the water body or onto land. The removal of dyes from industrial effluent is becoming a major problem for the textile industry as Indian government legislation is becoming more stringent. Wet processing units around textile cities were closed due to a lack of effluent treatment plants. Water is one of the basic requirements for the nutrition and continuation of life. Wastewater is difficult to view large-scale pollution caused by industrial, agricultural, and domestic activities. These activities produce wastewater that contains both organic and inorganic pollutants. A high level of COD and color are present in the effluent textile plants. A plant produces an amount of 250 mg/hour of polluted water. The present wastewater treatment schemes on the site of the plant consist of screening, pre-neutralized, anaerobic lagoon, post-neutralization, activated sludge process and sedimentations.

The activated carbon (AC) is associated with from the times of Ancient Egypt (1500 BC). Activated carbon is known as activated charcoal which is a form of carbon processed to small, low-volume pores that increase the surface area available for adsorption or chemical reactions. The surface area of activated carbon is more than 3000 m^2 which is determined by gas adsorption. The gas is attracted to the carbon material via Van der Waals forces and it is used in methane and hydrogen storage, air purification, solvent recovery, gold purification, metal extraction, water purification, medicine, sewage treatment, air filters in gas masks, and respirators, filters in compressed air, teeth whitening, production of hydrogen chloride and many other applications. Nowadays the AC (activated carbon) has been used in industries as a decolorizing agent. The charcoal is used in medical facilities such as in filtering masks for lab technicians, in kidney and liver dialysis machines, and even as markers in breast cancer surgery. The charcoal is not only used for removing toxins ingested by humans, but also used for a veterinarian’s pet that may have ingested something potentially harmful to them. During wars, respiratory protective devices were developed as personal protection tools against the use of toxic gases.

Activated carbon adsorption has been cited by the US Environmental Protection Agency (USEPA) as environmental pollution control technology. For adsorption, activated carbon is cost-effective. Activated carbon can be manufactured from any material that possesses reasonable elemental carbon content. Some of the precursors for activated carbon such as bagasse, scrap tires, sawdust, almond, pecan, english walnut, black walnut, macadamia nut, pistachio hazelnut shells, rice husk, rice bran, coal, peat, coconut shell, and other inexpensive materials with high carbon content (Pongener *et al.*, 2015). Because of their high absorptive capacities, this activated carbon can be used to remove organic and inorganic contaminants from water or air. The presence of surface oxygen groups increases the absorption properties of activated carbon, thus enhance the absorption of cations and anions from aqueous solution.

Commercially known coal-based activated carbons are most commonly used as an adsorbent for the removal of dye effluent due to their high adsorption capacity and high surface area (Gercel *et al.*, 2007). Because of its porous structure and high surface area, it is also finding applications in chemical and petrochemical industries, production of capacitors, etc. The chemical or physical methods can be used to activate the carbon to remove the effluent. The chemicals mainly involved are H_3PO_4 (El-Sayed *et al.*, 2014), H_2SO_4 (Karagoz *et al.*, 2008), HNO_3 (Pongener *et al.*, 2015), zinc chloride (Cronje *et al.*, 2011), KOH (Xiao *et al.*, 2012). The carbon content of the pressure, the nature and concentration of chemical activator, impregnation time, and pyrolysis temperature is playing a vital role in the characteristics of resulting activated carbon (Arriagada *et al*., 1997 and Sanchez *et al.*, 2001).

The activated carbon production of agricultural by-products has potential economic and environmental impacts. Firstly, it converts agricultural residue waste to high-value adsorbents. Secondly, activated carbons are widely used in water to remove organics, chemicals, and metals from the environment. Finally, it will reduce the import of activated carbon, as a result, increasing our economic base of the country (Ekpete *et al*., 2017). At present physical, chemical, and biological methods are generally used as a treatment process for removing dye effluent from textile wastewater (Rahman *et al.*, 2013).

Reports are available for the preparation of activated carbon from various agricultural residues like sugarcane bagasse (Fabon *et al.*, 2013), wood (Hared *et al.*,

2007) coconut shells and palm shells (Daud and Ali, 2004), lignin (Carrott and Carrott, 2007) and rice husk (Rahman and Bari, 2011).

Preparation of activated carbon using chemical and physical activation has been widely studied using various low-cost biomass materials such as coconut shell, nutshell, and apple pulp, to name a few (Laine and Yunes, 1992; Toles *et al.*, 1997 and Suárez-García *et al.*, 2001).

Activated carbon is a broad-spectrum agent that effectively removes toxic and refractive substances such as insecticides, herbicides, chlorinated hydrocarbons, and phenols, typically present in many water supplies (Bagreev *et al.*, 2001). Activated carbons are extremely porous carbon that has been treated to increase its adsorption performance.

The impregnation of rice precursors with KOH or NaOH enhances the surface area. Besides the activation temperature can be lowered. Washing rice straw with alkaline solutions like NaOH allows reducing the ash content (Huang *et al.*, 2001). Activated carbon demand will benefit from a continuing intensification of the global environmental movement as well as rapid industrialization. There are two different processes for the preparation of AC: physical activation and chemical activation. Physical activation involves gasification of the char oxidation steam, carbon dioxide, air, or any mixture of these gases in the temperature range from 800 to 1100^0C. In chemical activation, the carbonization and activation are accomplished in a single step by carrying out the thermal decomposition of the raw material impregnated with certain chemical agents. The advantage of chemical activation has low energy cost due to lower temperature (500-800^0C) than those needed for physical activation and high product yield.

In this study different types of oil cakes such as Almond, Sesame, Mustard, Coconut and Rice Bran, Cotton, Mustard, Neem, Peanut, and Boiler ash were used for the preparation of activated carbon and it was compared with Commercial activated carbon. Six activated carbons were successfully prepared by the activation process. Oil cakes are the residues obtained after oil is removed from various oil seeds. It is rich in protein and minerals and can be used as animal and poultry feed.

The oil cakes such as Almond, Rice bran, Sesame, Coconut, and Mustard, activated carbon were prepared by physical activation at 400˚C, followed by chemical activation by impregnating 2.5 N H_3PO_4, 1 N H_3PO_4, and 2.5 N H_2SO_4 in 1:5 ratios for overnight and it is then followed by physical activation at 300˚C for 2 hrs in a muffle furnace. The particle size of the activated carbon was further reduced by ball milling in Pulverisette 6 classic line (Fritsch, Russia) for 1 hour. The filtered activated carbon was also used for this study. Rice bran, due to its high cellulose and lignin content, can be used as a source of carbons to produce activated carbon. Coconut oil cake constitutes 20% fiber and 10% protein and de-oiled cake is used to make mixed cattle feed as well as to prepare char. Sesame oil cake contains 32% crude protein (CP), 8-10% oil, and total oil and albuminoids of 40-42%. In almond oil cake lipid fraction contained five fatty acids and activated carbon can be easily produced. Mustard oil cake is a 100% organic oil cake and can be the best source of activated carbon.

Wool fiber is the natural hair grown on sheep and is composed of a protein substance called keratin. Wool is composed of carbon, hydrogen, nitrogen and this is the only animal fiber, which contains sulfur also. Wool is the most reusable and recyclable fiber on the planet. Wool fiber has excellent absorbency, elasticity, resiliency, and moisture regain are high. Since wool is a protein fiber, the dyes are suitable for wool fiber.

The textile acid dyes are used to dye natural protein fibers such as wool, silk, nylon, and modified acrylics. An acid dye is a dye that is applied to textile at low pH. It contains sulphonic acid groups which are usually present as sodium sulphonate salts. These increases solubility in water and give the dye molecule a negative charge. Acid dye affix to fibers by hydrogen bonding, Van der Waals forces, and ionic bonding. Acid dye with deep and bright colors was selected for their high quality, fastness properties, their total dye penetration & high dye bath exhaustion. These are applied to the fibers from dye baths in acidic and neutral conditions. The dyeing is carried out at boiling temperature for 30-60 minutes, depending upon the depth of the shade and dyestuffs used.

The metal complex dyes are also called metalized dyes in which one or two dye molecules are coordinated with a metal ion. The dye molecule is typically a mono structure containing additional groups such as hydroxyl, carboxyl, or amino. These are capable of forming a strong coordination complex with metal ions such as

chromium, cobalt, nickel, and copper. Metal complex dyes are a combination of two types, one with 1:1 metal complex dye and another one with 1:2 metal complex dyes. These are water-soluble dyes and used for wool, silk, polyamides, and leather. They exhibit good fastness properties towards exposure to light. The fastness is depended upon the fiber type and dye type.

Very few studies have been conducted on the textile dye effluent. Considering the importance of the removal of dye effluent, a research work entitled “Removal of residual Textile dye effluent using activated carbon prepared from agricultural residues” has been planned with the following objectives:

1. Selection of dye effluent
2. Selection and preparation of adsorbents
3. Characterization of adsorbents
4. Standardization process of color removal

CHAPTER - II

REVIEW OF LITERATURE

Review of literature highlights the findings of related studies and eliminates the possibility of un-necessary duplication of efforts. It involves locating, reading and evaluating reports of research, reports of casual observations and opinion related to the individual's planned research project. It provides an opportunity to the researcher to know what has been done previously and what still remains to be done in the area. Review of literature helps to researcher in identifying the theoretical framework and methodological issues relevant to the study. Thus, the review of literature forms the foundation upon which all future research will be built. A comprehensive review of relevant literature is imperative in any scientific investigation, which designed to identify related research, to set the current research project within a conceptual and theoretical context. The present investigations have been reviewed under following subheadings;

2.1 Dye effluent in textile industry

2.2 Preparation of activated carbon

2.2.1 Physical reactivation

2.2.2 Chemical activation

2.3 Activated carbon use in removal of dye effluent

2.1 DYE EFFLUENT IN TEXTILE INDUSTRY

Yaseen and Scholz (2018) conducted study on textile dye wastewater characteristics and constituents of synthetic effluents found that textile wastewater contained various types of chemicals and contaminants at multiple ranges, which might be toxic to the specific microbes or plants in the treatment system due to its chemical toxicity and higher concentration of these compounds. The authors also suggested ingredients for a new mixture to simulate textile effluents.

Suresh (2015) reported that textile mill discharges contained bleaching chemicals, total dissolved solids (TDS), odor, total Kjeldahl nitrogen, suspended

solids (SS), salts, surfactants, heavy metals, mineral oils and high concentration of biological oxygen demand (BOD) and chemical oxygen demand (COD). The author also found that the existence of color in wastewaters was due to the discharge of unfixed dyes which after hydrolysis were incapable of reacting with the fabrics and discharge of untreated textile mill effluents known caused adverse impacts on the environment.

Elango *et al.* (2017) studied on the quality of textile dyeing effluent by analyzing the physico-chemical parameters such as colour, pH, biological oxygen demand (BOD), chemical oxygen demand (COD), total dissolved solids (TDS), chlorides, sulphides, calcium, iron, oil and grease of the effluent reported that all the parameters were found to be present in higher concentration than the standards given by BIS except for calcium, sulfide, and iron which implies that the effluents were toxic in nature and needed imperative treatment before disposal on water bodies to create pollution less and Eco-friendly environment.

Antoni *et al.* (2017) carried out work on the impacts of the textile industry discharges observed that large amount of highly colored wastewater and chemical reagents were present in the effluent. The author reported that among the alternative treatments, chemical methods like ozonation and Advanced Oxidation Process (AOPs) were found to be the best options to remove pigment and vat dyes in effluent.

Kritikos *et al*. (2007) conducted a study on textile manufacturing process and mentioned that it was apparent that textile wastewater must be treated, although the concentration of dyes in wastewater has been regularly lower than the other chemicals present. They frequently received prevalent concentration due to their strong color and it was highly visible even at very low concentration, therefore causing serious aesthetic and pollution problems in wastewater disposal and water transparency and gas solubility in lakes, rivers and other water bodies.

Akpen *et al*. (2017) conducted a study on removal of color from textile wastewater by mango seed endocarp activated carbon found that mango seed endocarp activated carbon (MSEAC) produced impregnation ratio of 1:4 which was the best among the impregnation ratios and the color removal was increased with decrease in initial color concentration. It was recommended that MSEAC can be used

effectively to remove color from textile wastewater at the optimum conditions thus converting the waste to wealth.

Chafidz *et al.,* (2018) while working on removal of methyl violet dye via adsorption using activated carbon which was prepared from *Randu* sawdust (*Ceiba pentandra*) found that the randu sawdust activated carbon possessed larger pore and surface area than the randu sawdust carbon/char. The optimum adsorption was recorded at pH 9 with 90 minutes of contact time and the maximum adsorption capacity of methyl violet dye by the randu sawdust activated carbon was recorded at 531.16 mg g^{-1}.

Singh *et al.,* (2003) worked on removal of color from wastewater using low-cost activated carbon prepared from agricultural waste material found that the methylene blue adsorption occurred through a film diffusion mechanism at low as well as at higher concentrations while methyl orange adsorption occurred through film diffusion at low concentration and particle diffusion at high concentrations and the adsorption of the two dyes increased with an increase in the temperature. The removal of both of the dyes was 100% at low concentration while the same decreased with an increase in the concentration. They revealed that the derived activated carbon could be fruitfully employed as an adsorbent for dye removal.

Ahmadi *et al.* (2020) while working on acid dye removal from aqueous solution by using neodymium (iii) oxide nanoadsorbents opined that optimal conditions at pH 3.15, AB92 concentration 138.5 mg/L, Nd_2O_3 nanoparticles dosage 0.83 g/L, and contact time at 49.55 min gave 90.70% adsorptive removal of AB92.

Mourid *et al.,* (2017) worked on activated carbon as the adsorbent for the removal of textile dye, acid green 1 (AG1) from aqueous solutions found that the adsorption capacity increased in the acid solutions, but decreased in the basic solutions with an optimum pH of 2 which was favored by the existence of positive charges on the surface of our material in an acid medium. The removal of AG1 by activated carbon is totally 100% at optimized conditions and the maximum adsorption capacity was reached 121.5 mg/g.

Singh *et al.,* (2005) worked on removal of Cr (VI) from wastewater using rice bran found that the maximum removal of Cr (VI) was found to be 99.4% at pH 2.0, initial Cr(VI) concentration of 200 mg l^{-1}, and temperature 20 °C.

Kebede and Gashaw (2017) worked on removal of chromium and azo metal-complex dyes using activated carbon that was synthesized from tannery wastes found that the bulk density, moisture content, volatile matter as well as ash content of the fleshed tannery waste activated carbon (FTWAC) showed good result which is 0.61 g/cm3, 4, 41 and 40% respectively. The FTWAC removed over 88% of test and odor for tannery effluents by the phenol number method. The FTIR showed that the active site had acidic functional group and the surface area that was characterized in BET instrument was 535.02 m^2. The maximum removal efficiency and adsorption capacity were recorded 99% and 7.8 mg/g for Cr (VI) at pH 3, 2 gram dose and 120 minutes of contact time respectively. Moreover, mordant black11(MB-11) and red azo dyes (AD) were showed maximum removal efficiency obtained, i.e. 97% for MB-11 at pH 9 and 93% of red AD at pH 6 with an optimum initial concentration was 120 ppm for MB-11 and 80 ppm for red AD. Furthermore the optimum contact time for Cr (VI) and both dyes were recorded as 120 minutes.

Razi *et al.,* (2018) carried out study on the removal of heavy metals from textile wastewater using sugarcane bagasse activated carbon found that sugarcane bagasse activated carbon (SBAC) found to be efficient adsorbent for the removal of metal ions. It was proven that the SBAC had performed 91 and 89% of metal removal (Fe and Zn respectively) due to the high surface area.

2.2 PREPARATION OF ACTIVATED CARBON

Ekpete *et al.* (2017) conducted a study from the preparation of activated carbon that was obtained from plantain (*Musa paradisiaca*) fruit, stem to study their characteristic properties found that CPPAC (carbonized plantain phosphoric acid activated carbon) was more preferable for adsorption than CPZAC (carbonized plantain zinc chloride activated carbon) which was due to its lower bulk density and ash content and high in iodine value.

Mutegoa *et al.,* (2014) conducted a study on preparation of activated carbon with desired properties through optimization of impregnating agent opined that the peanut shell activated carbon was given higher iodine number adsorbed at impregnation ratio 1:2 while sugarcane bagasse had provided higher iodine number adsorbed at impregnation ratio of 1:1. The higher number of iodine adsorbed by one gram of activated carbon was characterized with large number of micropore due to

higher carbon content, low ash content, high density and sufficient volatile content. In this condition, it seemed to be best suited for sugarcane bagasse than peanut shell and hence was suitable for the treatment of waste water associated with carboxylic acids.

Razi *et al.,* (2017) studied on the factors that affecting the removal of textile dye using adsorbent prepared from activated carbon reported that the low pH value was preferred for anionic dye whereas high pH values for cationic dye. For the adsorbent dose, the adsorption capacity increased along with the addition of adsorbent dosage due to the increase in the amount of sorption site. The contact time between adsorbent and dye adversely affect the efficiency of removal of dye where strong attraction of force shortened the time. In case of effect of dye initial concentration, with an increased in the initial concentration enhanced the adsorbent surface area to adsorb more dyes.

Girgis *et al.,* (2007) studied on X-ray diffraction patterns of activated carbons from sugarcane bagasse prepared under various conditions found that steam pyrolysis activated carbons exhibited bands around $2\theta = 23$ and 43° respectively, and XRD patterns of H_3PO_4 activated carbons displayed more developed and separated peaks in the early region with maxima at $2\theta = 23$, 26 and 29 respectively. Diffraction within $2\theta = 43°$ was broad, although depressed and diffuse suggesting that the intragraphitic layers were less developed.

Govardhan *et al.,* (2016) carried out study on agro industrial byproduct mediated green synthesis of silver nanoparticles (AgNPs) using cottonseed oil cake (CSOC) extract found that the XRD profile of biosynthesized AgNPs showed diffraction peaks at (111), (200), (142), (220), and (311) corresponding to the reflections of face centered cubic structure of synthesized AgNPs. The FTIR spectrum of the biosynthesized AgNPs showed strong absorption peaks at 3299, 2918, 1653, and 1051 cm^{-1} assigned O–H, C–H, C=C and N–H functional group respectively.

Saputra *et al.,* (2019) while working on the effectiveness of activated carbon from coconut shell and activated carbon corn cobs to be used for Pb^{2+} ion for adsorption using solid-phase spectrophotometry found that the adsorbent combination between activated carbon from coconut shell and corn cob increased the capabilities and effectiveness of the absorption to adsorb Pb (II) ion in the simulations waste and the most optimum ratio from the comparison between coconut shell and corn cob was

ratio of 1:2 with the absorption ability to adsorb Pb (II) ion in the simulations waste was 86.78%.

Hassan and Ahmed (2011) studied on synthesis and characterization of activated carbon (AC) from Saudi Arabian dates tree's fronds wastes found that single point BET surface area for ACs prepared via different concentration of H_3PO_4 indicated AC-60% showed the highest surface area of 1138 m^2g^{-1} compared to the raw date fronds of 4.6 m^2g^{-1} while slightly better to the commercial AC. The FTIR results showed chemical activation using H_3PO_4 successfully converted the raw material to pure activated carbon and showed similarities between commercial carbon and prepared carbon. SEM micrographs showed the well developed pores and cavity at AC-60%, which possessed high BET surface area.

Saeidi & Lotfollahi (2015) have reported a study that effect of powder activated carbon particle size on activated carbon monoliths properties. Methyl cellulose and bentonite were used to prepare activated carbon monolith (ACM) using powder activated carbon monolith (ACM) using powder activated carbon (PAC) by extrusion process. Powder activated carbon effects on porosity, adsorption and mechanical properties of the ACMs and four types of PAC including powder with particle size less than 150, 90, 50 and powder without screening were prepared and used. It was improved adsorption capacity, surface area and mechanical strength (both compression and impact strength) of the ACMs by screening the PAC micro particle particle sizes.

2.2.1 Physical reactivation

Tadda *et al.*, (2016) studied on activated carbon, its process, application and prospects and found that the activated carbon were fine and porous structure and had an extremely large particle surface area (>1000 m^2/g) that make it a powerful adsorptive properties. Hence, the adsorption process using activated carbon was found to be a potentially workable method of removing pollutants from aqueous solutions.

Buczek (2016) conducted a study on preparation of active carbon by additional activation with potassium hydroxide and to study the characterization of their properties found that 60.5% yield was obtained from the active carbon and the resulting carbon showed a well-developed porous structure with specific surface area

i.e., 2939m^2/g, total pore volume 1.488 cm^3/g, and micropore volume 1.001 cm^3/g respectively

Lan *et al.,* (2016) studied in preparation of activated carbon from Hawaii nut shell via steam physical activation and reported that high specific surface area of activated carbon was feasible by steam activation using Hawaii nut shells. Higher surface area mesoporous carbons were extremely favored liquid phase application. Removal of contaminants which might be in the gas or liquid phase may bring about huge economic benefits.

Chengyong *et al.,* (2018) conducted a study in progress in preparation and application of organic waste based activated carbon opined that organic waste can also be used for preparation of activated carbon such as agricultural waste. The author also suggested that research and development of new organic waste based activated carbon can improve the quality of activated carbon and can increase the yield and the proportion of activated carbon.

Sekirifa *et al.,* (2013) while working on preparation and characterization of an activated carbon from a date stones variety of physical activation with carbon dioxide found that the resulting product had a BET surface area ranged from 502 to 604 m^2/g and the ratio of micropore volume to the total pore volume ranged from 0.76 to 0.85. The obtained adsorption capacity lies between 23.25 mg/g and 28.57 mg/g. It was suggested that the variety of stone dates presented an interesting adsorptive proprieties and can be used as a source for activated carbon.

Sivakumar *et al.,* (2012) studied the physical, chemical and adsorption studies of activated carbon obtained from *Martynia annua L* and *Xanthiyam strumarium* reported that the high bulk densities and ash content was obtained from *Xanthiyam strumarium* compared to *Martynia annua L*. The zero point charge value of H_3PO_4 when treated carbon X3 indicated the presence of oxygen functional groups on its surface. They also reported that chemically modified carbon gave excellent adsorbent for the basic, acidic, and dispersed dyes, but direct dye showed less affinity for adsorption. Carbon X4 and X2 found to be removed efficiently the Direct Congo red dye from its aqueous solution and they possessed higher surface area. Carbon M3 and X3 showed greater adsorption due to the presence of acidic functional groups in it. The carbon M1 and X1 showed a greater amount of absorption of Reactive Blue MR

dye. Moreover, the carbon M6 and X4 showed the highest removal of acid blue dye due to their higher surface area. From SEM analysis, M3, X3 and X4 possessed many pores in a honeycomb shape that were clearly present on the surface.

IkhtiarBakti and Gareso (2018) studied on characterization of active carbon prepared from coconut shells using FTIR, XRD and SEM techniques found that the FTIR result showed that the carbon retained some functional IR groups, functional groups (C-O) until the coconut shell was successfully converted to carbon. The XRD results confirmed the existence of several phases of crystals like graphite around the peaks of 36° and 44° and there were two wide diffraction peaks which can be interconnected with carbon and graphite content. The SEM result showed that the carbonization of pyrolysis and activation processes created porosity and a large surface area for absorption.

Turangan *et al.*, (2017) worked on coconut shell charcoal that was obtained through the pyrolysis of coconut shell to study their FTIR, SEM and XRD analysis, which showed that the character of coconut shell charcoal before and after activated using FTIR, SEM and XRD analysis tend to be polar with absorption bands -OH groups, C = C, C = O and C-O particle distribution was nearly similar/uniform, smooth and visible presence of pores on the surface of charcoal and was semi crystal with a hexagonal crystal structure.

Rao (2021) investigated on characterization studies on adsorption of lead and cadmium using activated carbon prepared from waste tyres indicated that the FTIR spectra showed that the adsorption peak of O-H stretching, vibration was shifted from 3900 and 3075 cm^{-1} for lead and the asymmetrical stretching vibration at 3900 cm^{-1} was shifted to 3675 cm^{-1} for cadmium. In SEM analysis, the pores were completely filled with the metal ions after the adsorption of lead and cadmium metals and the pores appeared to be smooth which indicated that the metal was adsorbed to the functional groups present in the pores. From the XRD analysis, the CI index for raw, lead and cadmium loaded activated carbon of waste tyres were found to be 48.91%, 81%, and 54.9% respectively. The author found that increase in crystalline material present in the adsorbent after the adsorption of metal ions was due to the adsorption of metal ions onto the surface of the adsorbent.

2.2.2 Chemical activation

Rahman *et al.,* (2014) conducted a study on preparation of activated carbon by chemical activation and it's *in vitro* adsorption efficacy tests for paraquat reported that the paraquat was found to be adsorbed more by the produced activated carbon than the commercial activated carbon and the adsorbing capacity of paraquat activated carbon was increased with concentration. Both commercial and produced activated carbons were found to be effective for adsorption removal of paraquat.

Lamine *et al.,* (2014) studied chemical activation of an activated carbon that was prepared from coffee residue reported that the impregnation ratios had shown the capacity adsorption and surface area. The maximum adsorption capacity was recorded 5263mg/g and 5556 mg/g against activated carbon AC600-5 and AC700-12 respectively.

Heidarinejad *et al.,* (2020) studied methods for preparation and activation of activated carbon revealed that zinc chloride produced more surface area than phosphoric acid. Potassium carbonate produced higher yields with higher surface area for the adsorption of large pollutant molecules such as dyes as compared to potassium hydroxide. They also reported that when activating the carbon with potassium hydroxide the surface area and efficiency showed better results than sodium hydroxide. Moreover, in comparison with the physical mixing method and the impregnation method in activation with alkali metals, the activated carbon obtained through physical mixing showed higher porosity than the activated carbon produced by the impregnation method.

Mohammad *et al.,* (2015) studied the effect of phosphoric acid modification on characteristics of rice husk activated carbon found that the BET analysis showed that modification of the charred residue with phosphoric acid enhanced the surface area of the activated carbon from 12.47 to 102.4m^2/g. The average pore diameter was also enhanced from 2.4 to 1.82nm. It also showed improvement in micropore volume from 0.0052 to 0.034cm^3/g. Moreover, SEM analysis confirmed the improvement in surface area and pore development resulting from the phosphoric acid modification while FTIR analysis revealed the existence of phosphorous-oxy-containing functional groups on the surface of the phosphoric acid modified activated carbon.

Katiyar and Joshi (2015) studied on an agro-based waste material de-oiled mustard cake that was chemically activated with concentration H_2SO_4 to obtain an biosorbent for removal of water emulsified metal cutting fluids from aqueous medium/industrial wastewater found that adsorption was found to be less favorable with high influent concentration and maximum sorption capacity was obtained at low flow rate with large adsorbent dose.

2.3 ACTIVATED CARBON USE IN REMOVAL OF DYE EFFLUENT

Sharma *et al.* (2009) has used activated carbon developed from rice husk as the adsorbent for the removal of textile dye, acid green 1 (AG1) from aqueous solutions and wastewaters found that the AG1 removal increased from 93.75 to 94.91% by decreasing the initial concentration from 100 mg/l to 60 mg/l which can be concluded that the agro-waste rice husk can be successfully used as an adsorbent for removal of textile dye from aqueous solutions and wastewaters.

Karagoz *et al.* (2008) worked on laboratory prepared activated carbons from sunflower oil cake as adsorbents for the elimination of methylene blue (MB) from aqueous solutions found that the experimental impregnation ratio strongly affected the adsorption capacity. The maximum adsorption of MB onto the activated carbons was obtained at 25 °C and pH 6 for the activated carbons (AC1, AC2, and AC3) and the adsorption capacity for MB increased with increasing in the initial concentration of MB.

Deshpande *et al.* (2017) carried out work on removal of dye from aqueous solution by using activated carbon found that the coconut shell activated carbon (CSAC) gives better adsorption in the removal of methylene solution compared to the commercial grade activated charcoal (CGAC).

Okoniewska (2021) studied on removal of selected dyes on activated carbons found that the most effective adsorption under dynamic conditions was on ROW 08 Supra activated carbon. The longest time to complete depletion of sorption capacity obtained on this activated carbon was 76.5 h for tartrazine and 86 h for anilan yellow at a flow rate of 3 m/h. The author also found that an increase in filtration velocity resulted in a faster depletion of the adsorption capacity and shorter lifetimes of activated carbons were obtained.

Khatmode and Thakare (2015) studied on removal of pH, TDS, TSS and color from textile effluent by using sawdust as adsorbent found that pH was decreased from 7.9 to 7.2, the maximum percent removal for TSS was observed 45.50% and the maximum percent removal of TDS was observed 27% and the color of textile effluent changed from dark brown to light khaki.

Rao *et al.,* (2010) studied with the effect of sodium dodecyl sulfate (SDS) on the adsorption of Zn (II) and Ni (II) on carbon derived from mustard oil cake (CMOC) found that the addition of SDS to Zn (II) and Ni(II) solutions lead to a drastic increase in the adsorption capacity. The adsorption capacity for Zn (II) increased to 45.8 mg/g from 40 mg/g in the presence of SDS and that since Ni (II) adsorption capacity increased to 47.2 mg/g from 31 mg/g.

Robinson *et al.,* (2002) while working on removal of dyes from an artificial textile dye effluent using two agricultural waste residues, corncob and barley husk found that one gram (per 100 ml) of 600 mm corncob was found to be effective in removing a high percentage of dyes at a rapid rate (92% in 48 h) whereas one gram of 1 4 mm barley husk was found to be the most effective weight and particle size combination for the removal of dyes (92% in 48 h) which proved that barley husk was most effective biosorbents than corncob for the removal of textile dyes from effluent.

Popuri & Pagala (2019) carried out study on removal of dye from textile industry effluent by adsorption and coagulation methods observed that by adsorption technique with activated carbon that was prepared from saw dust, the highest color removal (787.04 %) was obtained in green color dye effluent at 90 RPM in 120 minutes with 2.5g dosage and pH 10.45 compared to coagulation and adsorption technique.

Saniya *et al.,* (2020) studied on removal of crystal violet dye from textile effluent using *Murraya koenigii* stem biochar found that the optimum operational parameters such as adsorbent dosage were found to be 100 mg l^{-1}, time (60 min), temperature (35°C) and dye concentration (50 mg l^{-1}). Moreover, the equilibrium adsorption of dye on the adsorbent was found to be 50 mg g^{-1} and the removal efficiency was about 70%. The characteristic studies of FTIR and SEM indicated the effective absorption of crystal violet onto curry tree carbon (CTC).

Igwegbe *et al.,* (2016) worked on adsorptive treatment of textile wastewater using activated carbon produced from *Mucuna pruriens* seed shells found that the removal of malachite green (MG) was found to be more spontaneous and feasible than the removal of Congo red (CR) on adsorbent *Mucuna pruriens* seed shells (AMSS). It was concluded that *Mucuna pruriens* seed shells activated with phosphoric acid were found to be a good adsorbent for the treatment of textile wastewater containing Congo red and malachite green.

CHAPTER - III

MATERIAL AND METHODS

This chapter deals with the description of research procedure technique used for experiment and analysis in the light of defined objectives. The research procedure followed has been categorized under the following sections:

3.1 Locale of the study
3.2 Selection of materials
3.3 Selection of dyes
3.4 Selection of adsorbent
3.5 Preparation of adsorbent
3.6 Selection of dye effluent
3.7 Characterization of adsorbents
3.8 Standardization of color removal parameters
3.9 Statistical analysis

3.1 LOCALE OF THE STUDY

The study was conducted in the Department of Textile and Apparel Designing, Faculty of Community Science, Assam Agricultural University, Jorhat, Assam, and Department of Textile Manufacturing and Textile Chemistry, Central Sheep and Wool Research Institute, Avikanagar, Rajasthan.

3.2 SELECTION OF MATERIALS

Wool fiber was used for the present study. Wool is a protein fiber which is derived from the fleece of sheep and other animals and it is composed of a protein substance called Keratin which is the major component of wool fiber. Raw wool contains 25-70% of impurities such as of wool grease, suint (sweat), dirt and vegetable matter such as sand and dirt. Vegetable matters are removed in worsted processing by carding and combing. The wool fiber contains 10% to 15% water and 35% of relative humidity. Wool fiber formed with 18 different types of amino acids. Some of them are glycine, alanine, valine, tyrosine, lysine, glutamic acid and cystine.

These amino acids have an amine group (-NH_2) at one end and an acid group (-COOH) at the other end. Wool fiber possesses good absorbency, elasticity, resiliency and high moisture regain. Wool is one of the most reusable and recyclable fiber.

3.3 SELECTION OF DYES

Dyeing is the application that is used to dye or pigments on textile materials such as fibers, yarns and fabrics. It is particularly done in a solution containing dyes and chemical fastness. Dye molecules are bind to the fiber by absorption, diffusion or bonding with temperature and time. Between dye molecule and fiber bonding it may be strong or weak which mainly depends on dyes. Dyes are composed of a group of atoms and are classified according to their application and chemical structure. They are known as chromophores, responsible for the color of a compound. The primary source of dye depends on nature where the dyes being extracted from plants or animals. For the present study, acid and metal complex dyes were used.

1. **Acid Dye: -** Acid dyes are extremely water soluble. They have better light fastness than basic dyes. Acid dyes are effective for protein fibers such as wool, silk, nylon and modified acrylics. They contain sulphonic acid groups, which are usually present as sodium sulphonate salts. The acid dye used for the study was ‘Optilan Turq’.

2. **Metal complex dye: -** Metal-complex dyes have low water solubility. The water solubility of metal-complex dye decreases from 1:1 to 1:2 and they shows good fastness when exposed to light and has moderate wet fastness properties. The metal-complex dye used for the study was ‘Lanagyn Brown’.

3.4 SELECTION OF ADSORBENT

Agricultural residues from field/agricultural processing wastes were used as adsorbent i.e. cotton, mustard, almond, peanut, rice bran, sesame, coconut, soyabean and neem oil cakes for the present study. The materials were collected from the different states of India i.e. Assam, Kerala, Maharashtra, Punjab, Kolkata and Rajasthan. Oil cakes are the residues obtained after oil is removed from various oilseeds. It is commonly used as animal feed.

3.5 PREPARATION OF ADSORBENT

The activated carbon was prepared from oil cakes. The following processes were used for preparing the activated carbon.

3.5.1. Process 1: The oil cakes were collected from different places of India and were dried in sun, broken with hammer and made into small pieces. The cakes were then crushed in a machine for mixing and made into powder. The powder of oil cakes were weighed 50g of each i.e. (coconut, cotton, peanut, soya, neem, rice bran, mustard, sesame, almond and boiler ash) and char was made by carbonization process. After carbonization the char was crushed properly and filtered through 140 nylon mesh. The acid activation process was used to prepare activated carbon. The chemicals of 1 N HNO_3 and 2.5 N HNO_3 was taken in a beaker and the char powder was added in the beaker. The solution was then boiled for half an hour through a heating mantal (applying heat to containers). After acid activation process the chemical was removed from the beaker and separate the powder for oven drying. After drying, the powders were then separated for 300˚C and 500˚C for calcinations in muffle furnace at temperature 425˚ C and 650˚ C respectively for 2 hours. After calcinations the powder was washed with distilled water until the pH comes to neutral. The powder was dried in oven and after drying the carbon was stored in an air tight plastic container. The dried materials were than weighed again.

3.5.2. Process 2: The oil cakes of coconut, cotton, peanut, soya, neem, rice bran, mustard, sesame and almond were dried in sun. The oil cakes were pulverized in a grinder and made into powder. Fifty grams of powder were treated with 1 N H_2SO_4, 2.5 N H_2SO_4, 1 N H_3PO_4 and 2.5 N H_3PO_4 for 24 hours in the rotary shaker. It was then squeezed through nylon mesh to remove excess acids and then kept in oven for 30 minutes for removal of moisture. Then the material was chared for carbonization. The weight of char was taken and then crushed to make it into powder form. It was then further subjected to calcination in muffle furnace at 300º C temperature for 2 hours. Activated carbon was then filtered through 140 nylon mesh size. The activated carbon powder was washed again to remove the excess acids (washing till neutral condition), kept in an oven for drying and then carbon was stored in an air tight plastic container. The dried materials were than weighed again.

3.5.3. Process 3: The oil cakes of almond, rice bran, sesame, coconut and mustard were dried under sun drying. The oil cakes were broken with hammer and made into small pieces. The material was then pulverized in a grinder. Three hundred grams of powder were added to 5% NaCl (on the weight of material) keeping the material to liquor ratio 1:25. The mixture was then kept under 200 rpm for 24 hour in the rotary shaker. After required time, the mixture was squeezed to remove the excess sodium chloride (NaCl). It is then subjected to acid activation using various concentrations of

1 N H_3PO_4, 2.5 N H_3PO_4, 1N H_2SO_4 and 2.5 N H_2SO_4 respectively for 24 hours in the rotary shaker with occasional stirring. After acidification the oil cakes were then squeezed and dried at 100°C to remove the moisture. The dried oil cakes were further chared and calcinated in muffle furnace at 300˚C for 2 h. The activated carbons, thus obtained were washed till it attains neutral pH, dried and yield was calculated. The powder was then filtered through 200 nylon mesh for the study. The particle size of the activated carbon was further reduced by ball milling in Pulverisette 6 classic line (Fritsch, Russia) for 1 hour.

Plate 3.1 (a): Almond oil cake

Plate 3.1(b): Sesame oil cake

Plate 3.1 (c): Rice Bran oil cake

Plate 3.1 (d): Coconut oil cake

Plate 3.1 (e): Mustard oil cake

PLATE 3.1: Raw oil cakes

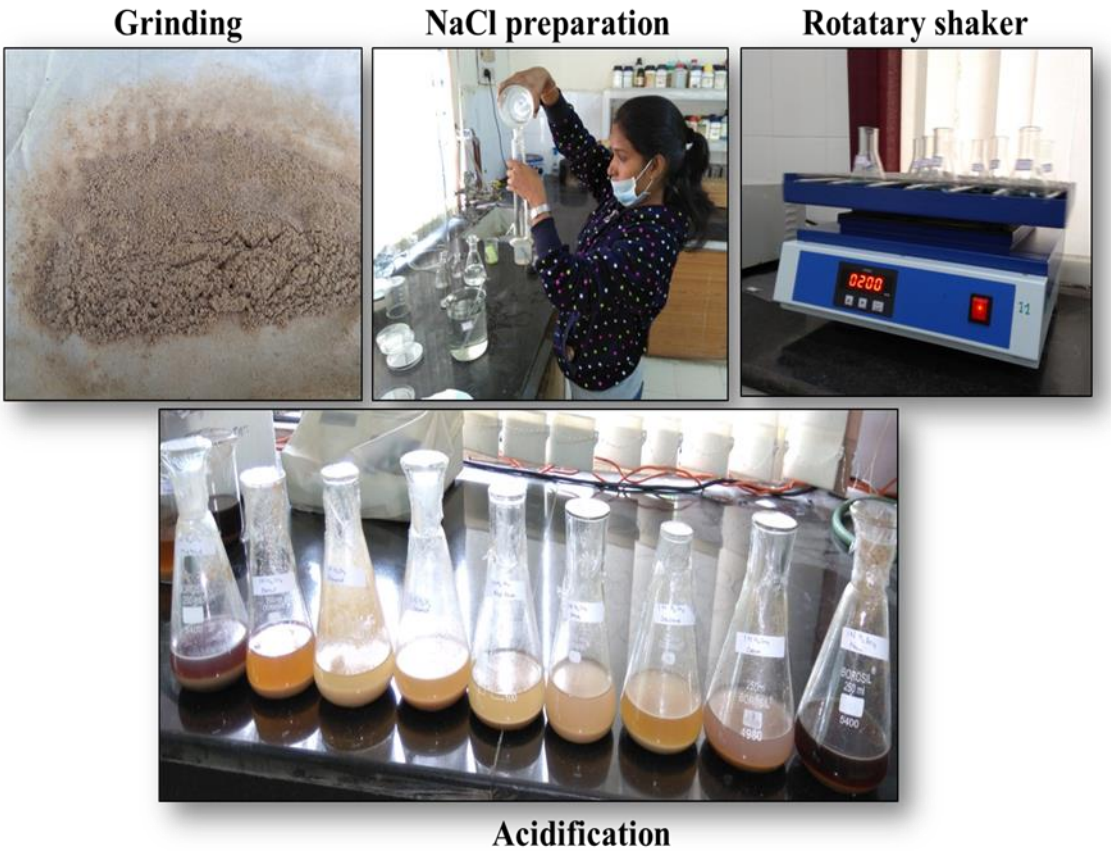


PLATE 3.2: Preparation of activated carbon

Plate 3.3 (a): 2.5 N H_3PO_4 Almond

Plate 3.3 (b): 1 N H_3PO_4 Coconut

Plate 3.3 (c): 2.5 N H_3PO_4 Mustard

Plate 3.3 (d): 2.5 N H_2SO_4 Almond

Plate 3.3 (e): 2.5 N H_2SO_4 Sesame

Plate 3.3 (f): 2.5 N H_3PO_4 Rice Bran

Plate 3.3 (g): Commercial activated carbon

PLATE 3.3: Prepared activated carbon

3.6 SELECTION OF DYE EFFLUENT

Wool fiber dyed with acid and metal complex and collected dye effluents were used for the present study.

3.6.1 Scouring:

Before dying, scouring was done for one kg wool fibers with ten litres of water, boiled and then added ten ml of ultravon JU (wetting agent) followed by twenty gram of sodium carbonate and mixed properly (Pandey *et al.,* 2017). Time was noted when the temperature was reached 80-85º C and after that wool was scoured for half an hour. Fiber was then rinsed thoroughly in water and squeezed in hydrometer machine and subjected to open air drying.

3.6.2 Metal complex dye:

Wool fiber dyed with 1:2 metal complex dye and their effluent was used for the present study. In a vessel, 9.2 liters of distilled water was taken. A dye solution was prepared with 45 g metal complex dye in 800 ml of boiled water. After that, solution was added into the vessel (having 9.2 litres distilled water) and the scoured wool fiber was put into it. 10 ml each of acetic acid and Ultravon JU were used for getting more dye uptake into the fiber. After reaching the temperature of dyeing solution at 90˚C it was then boiled for half an hour. The dyed wool fiber was left for 20-30 minutes for cooling at room temperature. After cooling, the wool fiber was squeezed thoroughly in hydrometer. An amount of 7.8 litres dye effluent was collected from the vessel and filtered it 2-3 times through 200 nylon mesh. Dye absorbency was recorded from effluent at 0.69 in photocolorimeter (Ladha, 2009).

3.6.3 Acid dye:

The same protocol was also used for the acid dye. An amount of 7.6 liters dye effluent was collected from the vessel and filtered it 2-3 times through 200 nylon mesh. Dye absorbency was recorded from effluent at 1.27 in photocolorimeter.

3.6.4 Preparation of dye effluent

The effluent used for the study was taken from the wool dyeing pilot plant at M/S Central Sheep and Wool Research Institute, Avikanagar, Rajasthan, India. The dyeing of wool with 1:2 metal complex dyes (Lanagyn Brown) was performed keeping 1:20 MLR and pH 7 using acetic acid. The dyeing was carried out at 90°C for

30 minutes without using any auxiliaries, except nonionic wetting agent (0.5 gpl). After dyeing, the dye effluent was collected, filtered and used for the experiment. The same protocol was followed for acid dye (Optilan Turq) also.

3.7 Characterization of adsorbents:

3.7.1 Surface area analysis: The surface area analysis of the biochar was performed using Nova Touc LX2 b Quantachromme instrument to obtain the adsorption isotherm and the surface area was calculated by Brunauer Emmett Teller (BET) method. The adsorbent of 1.5 g was mixed with 100ml dilution HCl at pH 3. 30 g of NaCl was added to it and the volume of the solution was made up to 150 ml with distilled water. The mixture was agitated for 5 minutes and then titrated with 0.1 N NaOH (Sodium Hydroxide), until the pH of the solution reaches to 9. The volume of NaOH consumed was recorded (Klank, 2006). The surface area was calculated by the following equation:

Before titration- After titration= final reading

3.7.2 FTIR analysis: FTIR analysis was performed for the identification of functional groups present in the activated carbon using Brooker double beam spectrophotometer (model- Alpha). For sample analysis, 0.1 g of activated carbon was taken and kept on a plate. The plate was then introduced into the spectrophotometer for analysis. The spectra were measured from 500-4000 cm^{-1}.

3.7.3 X-ray powder diffraction: The X-ray diffraction analysis was conducted using a Panalytical X'pert powder diffractometer and the resulted data were recorded.

3.7.4 Particle size analysis - The size of activated carbon was analyzed using the Malvern Mastersizer 3000 model particle size analyzer. Dispersion (0.1%) of activated carbon was prepared in water and sonicated before feeding to the particle size analyzer to prevent agglomeration. The wave length was observed and data was recorded.

3.7.5 SEM and EDX analysis: SEM and EDX analysis was performed using Nova Nano FESEM- 450 (Netherland) with suitable magnification. Scanning Electron microscopy was used for one pinch of adsorbent and kept in the machine. The picture with the clear image in the computer screen was taken for the results.

3.7.6 pH analysis: About 0.1 g of the adsorbent was added to 30 ml distilled water and boiled for 10 minutes. The solution was cooled at room temperature and filtered to remove the adsorbents. The pH of the solution was measured in pH meter and the values were recorded.

3.7.7 Bulk density: For the analysis of bulk density, a measuring cylinder of 10 ml was filled tightly with 3.0 g of adsorbents without any voids using a glass rod. The density was calculated from the following formula:

$$\text{Density} = \text{Mass}/\text{Volume}$$

Whereas, Mass = Total weight of the material

Volume = Total volume of the measuring cylinder

3.7.8 Porosity: Adsorbents of 1.5 g and 2.5 ml distilled water was taken in a 25 ml measuring cylinder. The volume of cylinder was recorded as V_1. It was kept for an hour and after one hour, again the volume was recorded (V_2). The porosity was calculated by the formula,

$$\text{Porosity } (\varepsilon) = V_2\text{-}W/V_1$$

3.7.9 Iodine number: Depending on the activated carbon 0.75 g of the dried carbon was weighed, 10 ml of 5% HCl solution was pipette out into the flask and swirled it until the activated carbon become wet. The flask was placed on a hot plate, the contents were brought to boil and was allowed boiling for exactly 30 seconds. The flask and contents were allowed to cool at room temperature, and then 100 ml of 0.1 N iodine solutions was added to it through pipette and the values were noted down.

Iodine solution: 40 g of potassium iodide was weighed in a 500 ml glass stopper flask and the volume was made up to 100 ml by adding distilled water. The solution was kept at room temperature. After that, 12.7 g of resublimed iodine (I_2) was added; the flask was restopped and swirled until the iodine was completely dissolved. The 50 ml sample was titrated with 0.10 N sodium thiosulphate solution until the yellow colour disappeared. 1 ml of starch solution was added and the titration was carried out until the blue colour indicator just disappeared. The volume of sodium thiosulphate solution used was recorded (AWWA 600-78). The iodine number was calculated by formula,

$$\text{Iodine value} = \frac{V_{(\text{initial value})} - V_{(\text{Final value})} \times (\text{Thiosulphate}) \times 126.9}{\text{Total solution in ml.}}$$

3.7.10 Ash content: For determination of ash content, crucibles were preheated to about 100˚ C, cooled in a desiccators, and the weighed 1.0 g of each sample was transferred into the crucibles and reweighed. The crucibles containing the samples were kept in the muffle furnace at 600°C for 6 h and was allowed to cool in the dessicator having zero humidity. From the difference in the weight, the ash content was calculated by following formula:

$$\text{Ash\%} = \frac{\text{Ash weight}}{\text{Oven dry weight}} \times 100$$

3.7.11 Zero point charge: The pH point of zero charge determination (pH_{pzc}) of the activated carbons were carried out by adding 0.1 g of activated carbon to 200ml solution of 0.1 M NaCl, and initial pH was checked. The volumetric flask were sealed and kept aside. The pH was checked after 16 hours and 24 hours respectively. The $pH_{(pzc)}$ occurs when there was no change in the pH after contact with adsorbent was recorded.

3.7.12 Moisture content: The adsorbent (0.5 g) was weighed in triplicayte and placed in clean, dried and weighted crucible. The crucibles were placed in an oven at 105º C for 6 hours. The moisture content of the adsorbent was analyzed from the difference between the weight before and after drying.

The calculation for moisture content on a wet-weight basis was done using the following formula:

$$\text{Moisture content (\%)} = \frac{W2 - W3}{W2 - W1} \times 100$$

Where,

W1 = weight of container with lid;

W2 = weight of container with lid and sample before drying; and

W3 = weight of container with lid and sample after drying

3.7.13 Methylene blue value: 1.2 g methylene dye was dissolved in 1000 ml standard measuring flask with the aid of 0.25% (V/V) acetic acid. The absorbance of the solution was then adjusted to 0.84 at 620 by dilution. Exactly 0.1 g of the activated carbon sample was added with 25 ml of the methylene blue test solution in a volumetric flask and was shaken until decolourization occurs. The addition of

methylene blue in the interval of 5 ml was continued till the saturation point, i.e., the colour persists. The amount of methylene blue added was noted. For each test, 3 replicates were taken for the study.

3.8 STANDARDIZATION OF COLOUR REMOVAL PARAMETERS

3.8.1 Colour value analysis: The colour absorbency of the untreated and treated effluent was measured using photocolorimeter/ UV VIS spectrophotometer. For metal complex dye and acid dye λ_{max} maximum absorbancy value was recorded at 620 and 490. The treated effluent was taken in equate and the absorbency was checked in photocolorimeter. The low absorbency value indicated that the color was removed from the treated effluent.

3.8.2 Adsorbent concentrations for Metal complex dye: Removal of metal complex dye effluent, adsorbent concentration at 0.1%, 0.25%, 0.5% and 1.0% respectively were taken with three replication for each concentration. For dye effluent treatment, 25 ml of dye solution was taken for each replica. Previously weighted adsorbents were then added and mixed thoroughly within a shaker for different time *viz.*, 30 minute, 60 minute and 90 minute respectively. The solution was filtered through filter paper and kept it for settlement of the particle of activated carbon and then absorbency was checked in photocolorimeter.

3.8.3 Adsorbent concentrations for Acid dye: Removal of acid dye effluent, adsorbent concentration at 0.5%, 1.0%, 1.5% and 2.0% were taken with three replication for each concentration. For dye effluent treatment, 20 ml of dye solution was taken for each replica. Previously weighted adsorbents were then added and mixed thoroughly within a shaker for different time *viz.*, 30 minute, 60 minute and 90 minute respectively. The solution was filtered through filter paper and kept it for settlement of the particle of activated carbon and then absorbency was checked in photocolorimeter.

3.8.4 Duration of treatment: The time duration for treatment *viz.*, 30, 60 and 90 minutes each with 3 replication at 150 RPM (Rotation per minute) were taken for removal of dye effluent and then the solution was filtered through filter paper.

3.8.5 pH of the effluent: pH of the effluent were taken at neutral condition. After effluent treatment pH was checked and different pH range was recorded against different treated dye effluent.

3.8.6 Total Dissolve Solution (TDS): The adsorbent concentration with three replications were taken, mixed together and treated for dye effluent to analyze the Total Dissolve Solution by TDS meter and the results were recorded in g/ml.

3.8.7 Biological oxygen demand (BOD): Adsorbents 2.5 N H_2SO_4 and 2.5 N H_3PO_4 almonds treated with 500ml and 1000ml solution of metal complex dye and acid dye effluent were taken. The temperature of the effluent was kept at 20°C for 5 days. The physico-chemical parameters of acid dye and metal complex dye effluent have been analyzed and the experimental results were compared with standard I.S 3025 (Part 44) and APHA 22nd edition (Method no. 5210B) 2012 respectively.

3.8.8 Chemical oxygen demand (COD):): Adsorbents 2.5 N H_2SO_4 and 2.5 N H_3PO_4 almonds treated with metal complex dye and acid dye solution of effluent i.e. 500ml and 1000ml were taken. The physico-chemical parameters of acid dye and metal complex dye effluent have been analyzed and the experimental results were compared with standard I.S 3025 (Part 58) and APHA 22nd edition (Method no. 5220B) 2012 respectively.

3.9 STATISTICAL ANALYSIS:

All the data obtained were expressed as Mean ± SD using Microsoft Office Excel 2007. Analysis of variance (ANOVA) in two-factor completely randomized design was performed on the data using Statistical Package for Social Science (2006) and the means were tested for significance at 5 percent probability level using Duncan's Multiple Range Test (DMRT) as provided in the same SPSS (2006).

If the value of the variance was found to be significant the standard error of the mean with different significant were found by calculating the critical difference (Sneckdecor and Cochran, 1979).

S.Ed± = √ (2× Error mean square/ No. of replications)

Significance of specific mean difference was determined by calculating the critical difference at 5 percent level of probability with the following formula:

Critical difference (CD) = S.Ed. (±)×'t'.

Where, 't' = tabulated value of't' at 5 percent level of significance at error degrees of freedom.

CHAPTER IV

RESULTS AND DISCUSSION

This chapter includes the findings and discussion about the color removal of dye effluent using activated carbon from agricultural residues and the results are presented under the following headings:

4.1 Characterization of activated carbon

I. Bulk density of activated carbons

II. Porosity of activated carbons

III. Ash content of activated carbons

IV. Moisture content of activated carbons

V. Methylene blue of absorbents

VI. pH analysis of activated carbons

VII. Zero point charge analysis of activated carbons

VIII. Particle size analysis

IX. Iodine number

4.2 SEM analysis of activated carbons

4.3 EDX analysis of activated carbons

4.4 Chemical Oxygen Demand

4.5 Biological Oxygen Demand

4.6 XRD analysis of activated carbon

4.7 BET analysis of activated carbon

4.8 FTIR analysis of activated carbons

4.9 Absorbency of Acid dye effluent

4.10 Absorbency of Metal complex dye effluent

4.11 pH of treated sample with acid dye

4.12 pH of treated sample with metal complex dye

4.13 TDS of treated sample with acid dye

4.14 TDS of treated sample with metal complex dye

4.1 Characterization of activated carbon in acid dye

4.1.1 Characterization of activated carbon in acid dye using 2.5 N H_3PO_4 Almond activated carbon

Table 4.1: Characterization of 2.5 N H_3PO_4 Almond activated carbon

Activated carbons	Treatment	Bulk Density (g/cm^3)	Porosity (%)	Ash content (%)	Moisture content (%)	Methylene blue (mg/g)	pH	Zero point charge (pzc)	Particle size analysis (nm)	Iodine number (m^2/g)
2.5 N H_3PO_4 Almond	Filtered	4.400	3.533	0.120	0.048	109.000	5.963	4.067	652.333	20.833
	Ball milling	4.533	4.500	0.176	0.040	123.667	6.180	4.140	310.333	22.067
	Commercial activated carbon	6.467	6.533	0.118	0.117	488.000	5.343	5.750	447.667	10.000
	S. Ed(±)	**0.1678**	**0.1981**	**0.0183**	**0.0008**	**1.5154**	**0.1310**	**0.0908**	**1.5635**	**5.5246**
	$CD_{(0.05)}$ for activated carbon	**0.4105**	**0.4848**	**0.0447**	**0.0020**	**3.7079**	**0.3206**	**0.2223**	**3.8257**	**NS**

Foot note: - Data given are mean of three replications.

The finding of different characteristics of 2.5 N H_3PO_4 almond was exhibited in the table 4.1 and figure 4.1 (a-i), from the table it is clear that some of the characteristics of 2.5 N H_3PO_4 almond activated carbon such as bulk density, porosity, ash content, methylene blue, pH, zero point charge, and iodine number showed the decreasing trend while particle size and moisture content recorded an increasing trend in case of filtered activated carbon. Moreover, particle size and moisture content were decreased in respect of ball milling activated carbon. From the statistical analysis, it was clear that all the properties except iodine number found to be significant at 5% level with respect to 2.5 N H_3PO_4 almond activated carbon.

Bulk density recorded in commercial activated carbon was (6.467g/cm^3) but in 2.5 N H_3PO_4 almond (ball milling) activated carbon recorded as (4.533g/cm^3) and in 2.5 N H_3PO_4 almond (filtered) activated carbon it was recorded as (4.400g/cm^3). The porosity exhibited in commercial activated carbons was (6.533%), ball milling (4.500%), and in filtered (3.533%) respectively. Ash content found in commercial activated carbon as (0.118%), ball milling (0.176%), and filtered (0.120%) respectively. The moisture contents were listed in commercial activated carbon as (0.117%), ball milling (0.040%), and filtered (0.048%) respectively. The methylene blues found in commercial activated carbons were (488.000mg/g), ball milling (123.667mg/g), and in filtered (109.000mg/g) respectively. The pH of the activated carbon exhibited in commercial activated carbons was (5.343), ball milling (6.180), and filtered (5.963) respectively. The zero point charges of the activated carbon were recorded (5.750pzc) in commercial activated carbon, (4.140pzc) in ball milling and (4.067pzc) in filtered respectively. The particle size analyses of the activated carbon exhibited in commercial activated carbon were (447.667nm), ball milling (310.333nm), and in filtered (652.333nm) respectively. The iodine number was recorded in commercial activated carbon were (10.000m^2/g), ball milling (22.067m^2/g), and in filtered (20.833m^2/g) respectively.

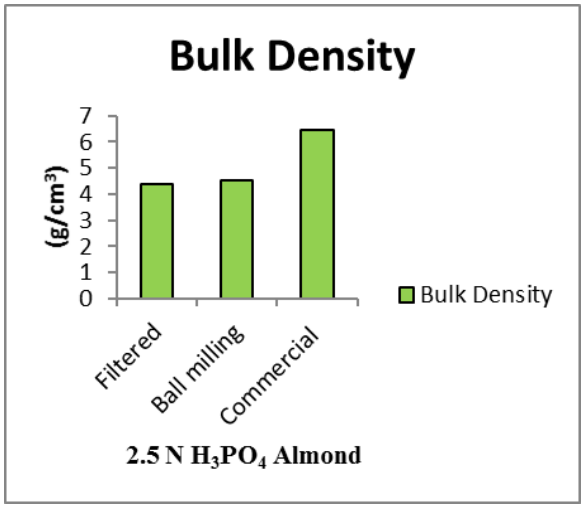


Figure 4.1 (a): Mean of bulk density

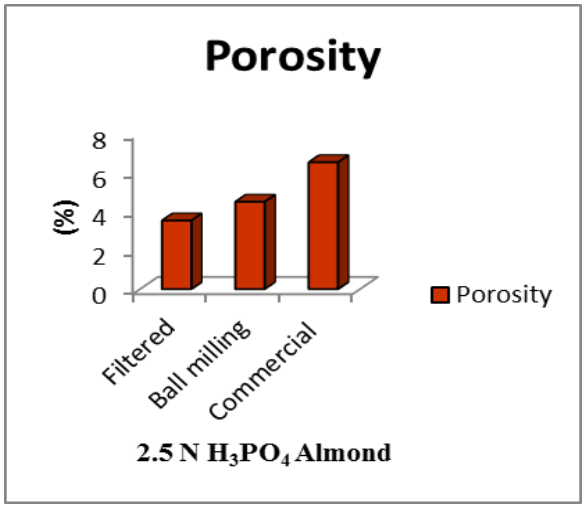


Figure 4.1 (b): Mean of Porosity

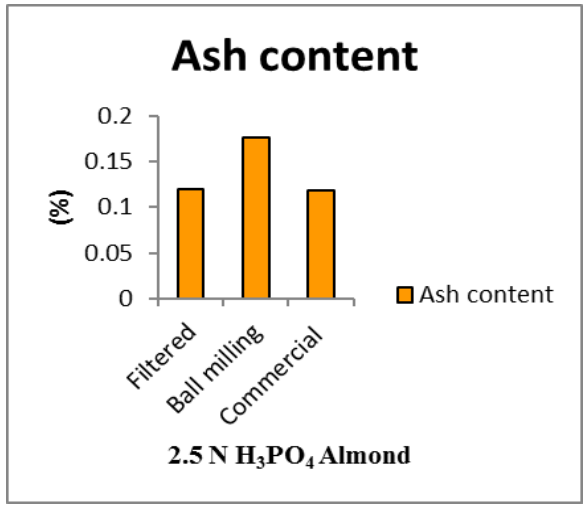


Figure 4.1 (c): Mean of ash content

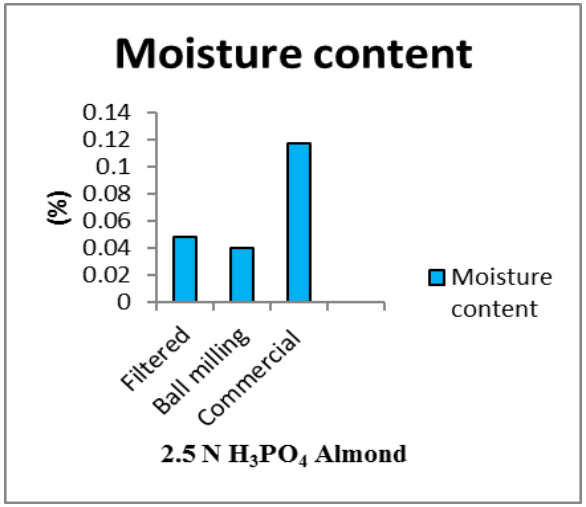


Figure 4.1 (d): Mean of moisture content

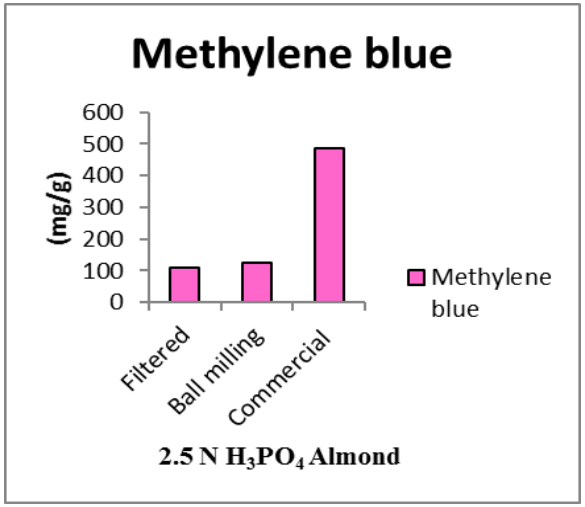


Figure 4.1 (e): Mean of methylene blue

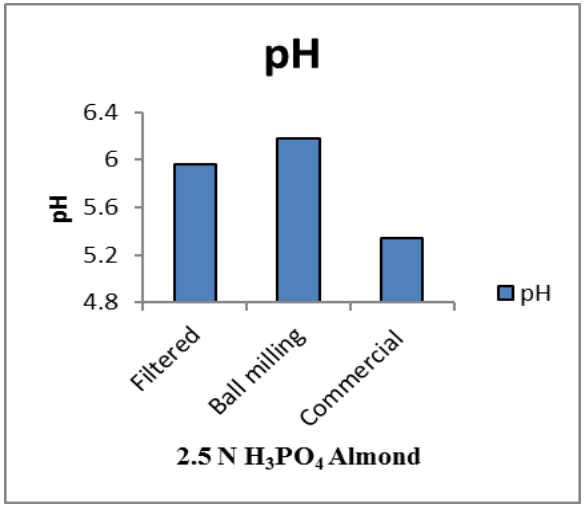
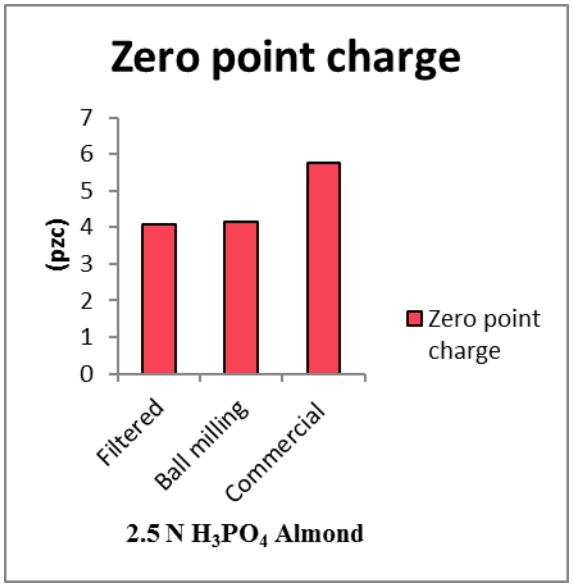
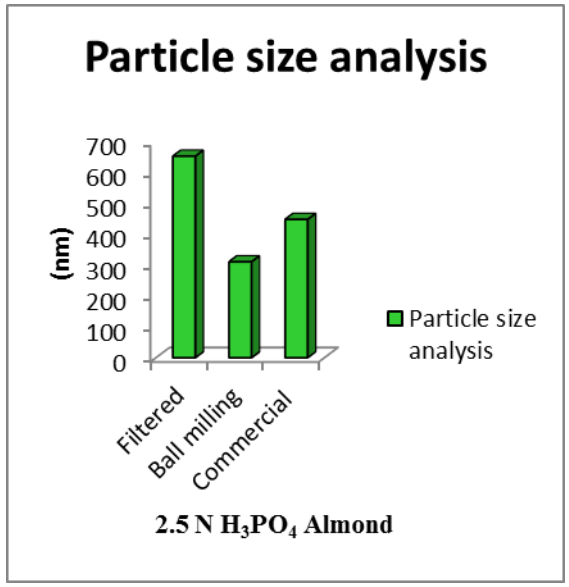


Figure 4.1 (f): Mean of ash pH





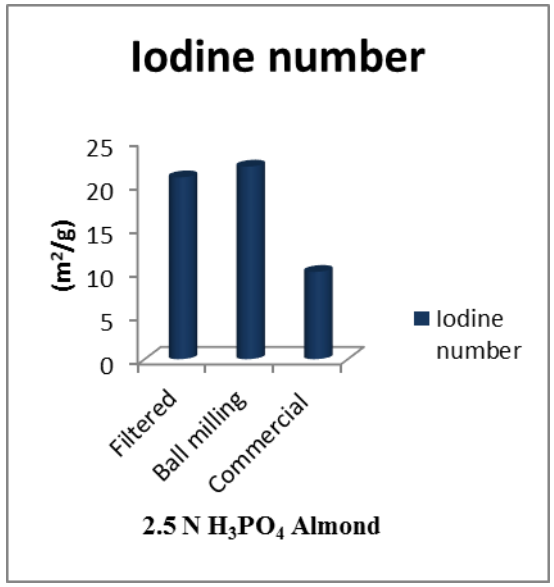


Figure 4.1 (g): Mean of zero point charge **Figure 4.1 (h): Mean of particle size analysis** **Figure 4.1 (i): Mean of iodine number**

Figure 4.1: Characterizations of 2.5 N H_3PO_4 Almond activated carbons

Saeidi and Lotfollahi (2015) worked on effects of powder activated carbon of monoliths to study the particle size found that with decreased particle size of powder activated carbon (PAC) led to dramatically improved adsorption capacity, surface area and mechanical strength of the produced activated carbon monolith (ACM).

4.1.2 Characterization of activated carbon in acid dye using 1 N H_3PO_4 Coconut activated carbon

Table 4.2: Characterization of 1 N H_3PO_4 Coconut activated carbon

Activated carbons	Treatment	Bulk Density (g/cm^3)	Porosity (%)	Ash content (%)	Moisture content (%)	Methylene blue (mg/g)	pH	Zero point charge (pzc)	Particle size analysis (nm)	Iodine number (m^2/g)
1 N H_3PO_4 Coconut	Filtered	4.500	4.367	0.214	0.085	148.333	6.480	4.180	842.667	16.667
	Ball milling	4.800	4.233	0.218	0.063	164.000	5.660	3.850	112.333	16.500
	Commercial activated carbon	6.467	6.533	0.118	0.117	488.000	5.343	5.750	447.667	10.000
	S. Ed(±)	**0.2018**	**0.1414**	**0.0082**	**0.0014**	**1.5154**	**0.0996**	**0.1908**	**1.8257**	**3.1366**
	$CD_{(0.05)}$ for activated carbon	**0.4939**	**0.3460**	**0.0200**	**0.0035**	**3.7079**	**0.2437**	**0.4668**	**4.4674**	**NS**

Foot note: - Data given are mean of three replications.

The table 4.2 and figure 4.2 (a-i), showed the characteristics of activated carbon 1 N H_3PO_4 of coconut. The result depicted that some of the characteristics of 1 N H_3PO_4 coconut activated carbon such as bulk density, ash content and methylene blue, showed the decreasing trend while, pH, zero point charge, iodine number, porosity, particle size and moisture content registered an increasing trend in case of filtered activated carbon. Furthermore, particle size, moisture content, pH, zero point charge and iodine number were decreased with respect of ball milling activated carbon. From the statistical analysis, it indicated that all the properties except iodine number found to be significant at 5% level with respect to 1 N H_3PO_4 coconut activated carbon.

Bulk density was recorded in commercial activated carbon as (6.467g/cm^3), ball milling (4.800g/cm^3), and in filtered it was recorded as (4.500g/cm^3) respectively. The porosity was listed in commercial activated carbon as (6.533%), ball milling (4.233%) and in filtered it was recorded as (4.367%) respectively. Ash content was recorded in commercial activated carbon (0.118%), ball milling (0.218%) and filtered (0.214%) respectively. The moisture content exhibited in commercial activated carbon was (0.117%), filtered (0.085%), and lowest value of moisture content was found in ball milling (0.063%). The methylene blue recorded in commercial activated carbon (488.000mg/g), ball milling (164.000mg/g) and in filtered (148.333mg/g) respectively. The pH was recorded in commercial activated carbon (5.343), ball milling (5.660), and filtered (6.480) respectively. The highest zero point charge was listed in commercial activated carbon as (5.750pzc), in ball milling (3.850pzc) and the filtered activated carbon showed (4.180pzc) respectively. The particle size analysis obtained in commercial activated carbon was (447.667nm), in ball milling (112.333nm), and in filtered it was recorded as (842.667nm) respectively. The iodine number found in commercial activated carbon was (10.000m^2/g), ball milling (16.500m^2/g), and in filtered it was exhibited as (16.667m^2/g) respectively.

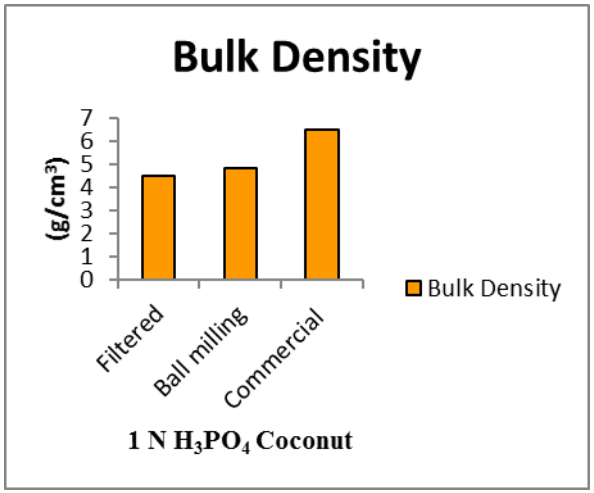


Figure 4.2 (a): Mean of bulk density

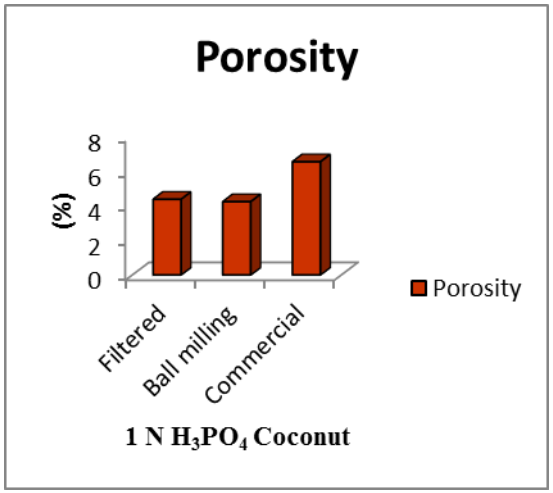


Figure 4.2 (b): Mean of Porosity

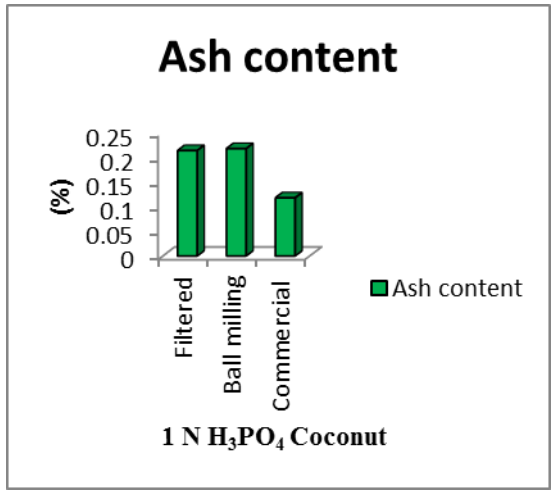


Figure 4.2 (c): Mean of ash content

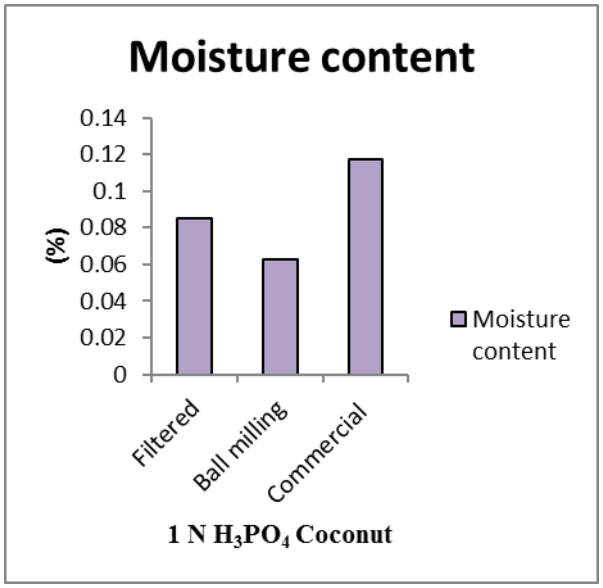


Figure 4.2 (d): Mean of moisture content

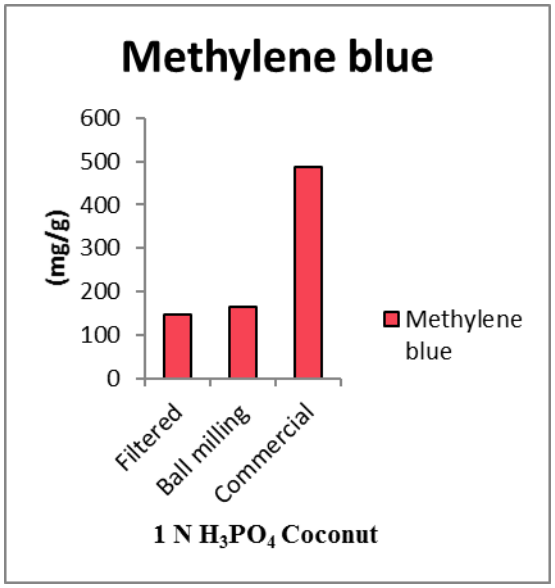


Figure 4.2 (e): Mean of methylene blue

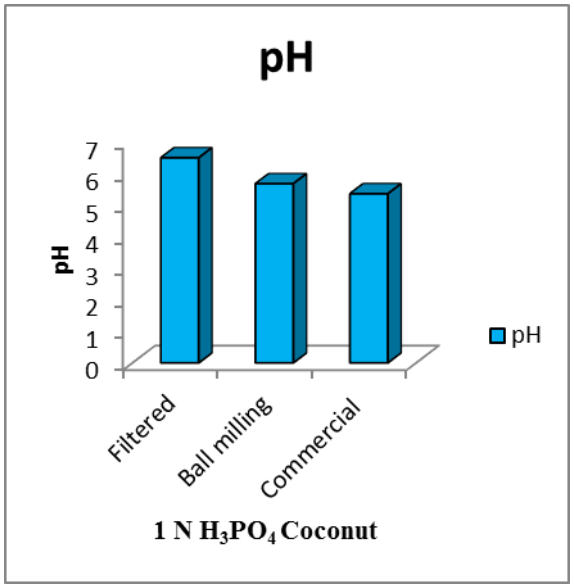


Figure 4.2 (f): Mean of ash pH

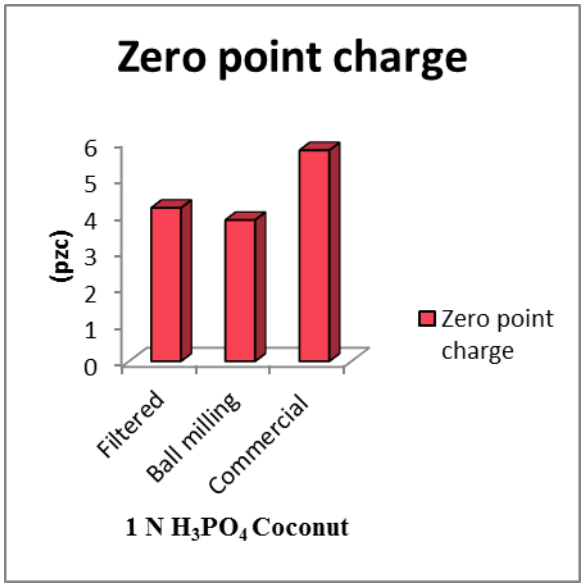


Figure 4.2 (g): Mean of zero point charge

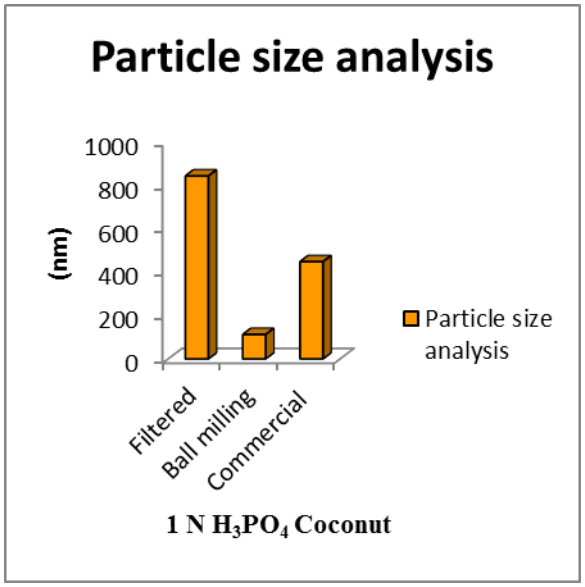


Figure 4.2 (h): Mean of particle size analysis

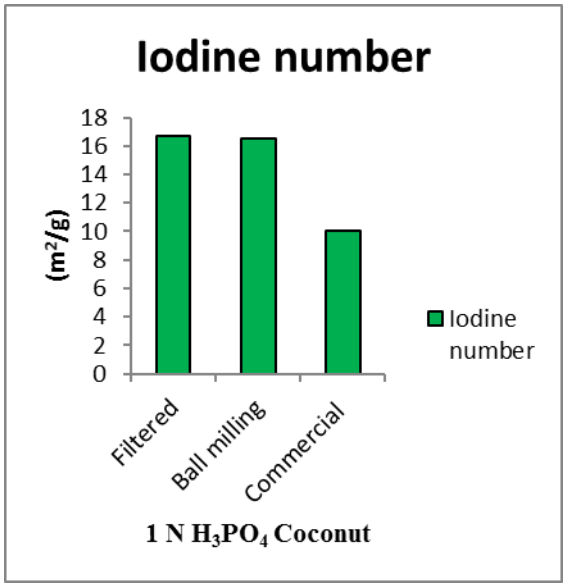


Figure 4.2 (i): Mean of ash iodine number

Figure 4.2: Characterizations of 1 N H_3PO_4 Coconut activated carbons

4.1.3 Characterization of activated carbon in acid dye using 2.5 N H_3PO_4 Mustard activated carbon

Table 4.3: Characterization of 2.5 N H_3PO_4 Mustard activated carbon

Activated carbons	Treatment	Bulk Density (g/cm^3)	Porosity (%)	Ash content (%)	Moisture content (%)	Methylene blue (mg/g)	pH	Zero point charge (pzc)	Particle size analysis (nm)	Iodine number (m^2/g)
2.5 N H_3PO_4 Mustard	Filtered	4.300	3.433	0.220	0.076	74.000	5.837	3.743	234.333	12.400
	Ball milling	4.567	4.567	0.213	0.082	125.333	6.100	3.647	142.333	14.733
	Commercial activated carbon	6.467	6.533	0.118	0.117	488.000	5.343	5.750	447.667	10.000
	S. Ed(±)	**0.1466**	**0.1700**	**0.0063**	**0.0008**	**1.5154**	**0.1306**	**0.0922**	**1.5635**	**2.4410**
	$CD_{(0.05)}$ for activated carbon	**0.3586**	**0.4159**	**0.0153**	**0.0020**	**3.7079**	**0.3196**	**0.2256**	**3.8257**	**NS**

Foot note: - Data given are mean of three replications.

The table 4.3 and figure 4.3 (a-i), showed the characteristics of activated carbon 2.5 N H_3PO_4 of mustard. The result depicted that some of the characteristics of 2.5 N H_3PO_4 of mustard activated carbon such as bulk density, porosity, moisture content, methylene blue, pH, zero point charge and iodine number showed a decreasing trend while ash content and particle size listed an increasing trend in case of filtered activated carbon. Besides, ash content and particle size were decreased with respect of ball milling activated carbon. From the statistical analysis, it was found that all the properties except iodine number found to be significant at 5% level with respect to 2.5 N H_3PO_4 of mustard activated carbon.

Bulk density was recorded in commercial activated carbon as (6.467g/cm^3), ball milling (4.567g/cm^3), and in filtered (4.300g/cm^3) respectively. Porosity registered for commercial activated carbon were (6.533%), ball milling (4.567%), and in filtered (3.433%) respectively. Ash content obtained in commercial activated carbon was (0.118%), ball milling (0.213%) and filtered (0.220%) respectively. Moisture contents were recorded in commercial activated carbon (0.117%), ball milling (0.082%), and filtered (0.076%) respectively. The methylene blue found in commercial activated carbon was (488.000mg/g), ball milling (125.333mg/g), and in filtered (74.000mg/g) respectively. The pH was recorded in commercial activated carbon (5.343), ball milling (6.100), and filtered (5.837) respectively. Zero point charges were found in commercial activated carbon (5.750pzc), ball milling (3.647pzc) and filtered (3.743pzc) respectively. The particle size analysis was recorded in commercial activated carbon (447.667nm), ball milling (142.333nm), and in filtered (234.333nm). The iodine numbers were exhibited in commercial activated carbon (10.000m^2/g), ball milling (14.733m^2/g) and in (12.400m^2/g) respectively.

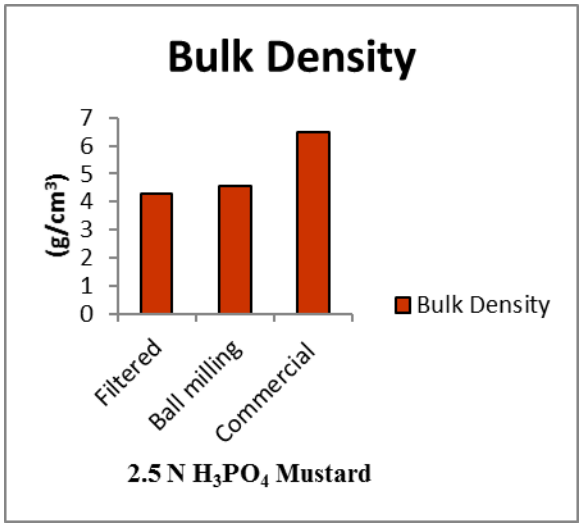


Figure 4.3 (a): Mean of bulk density

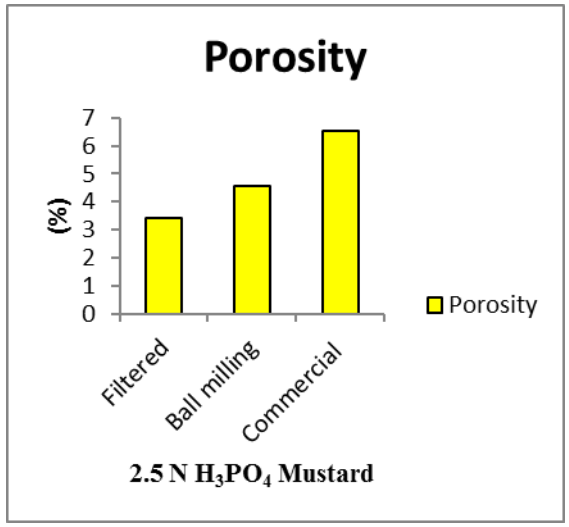


Figure 4.3 (b): Mean of Porosity

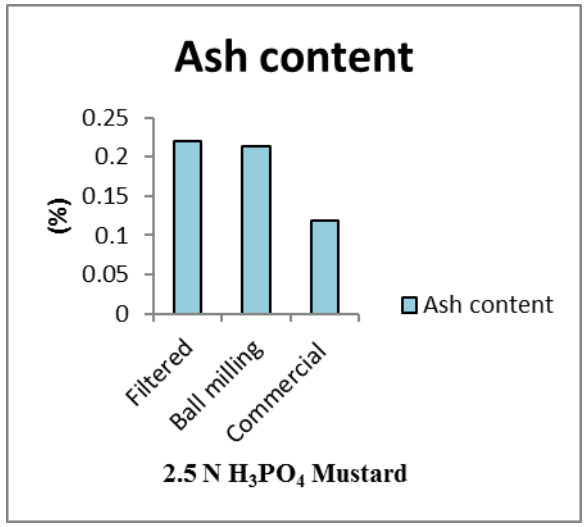


Figure 4.3 (c): Mean of ash content

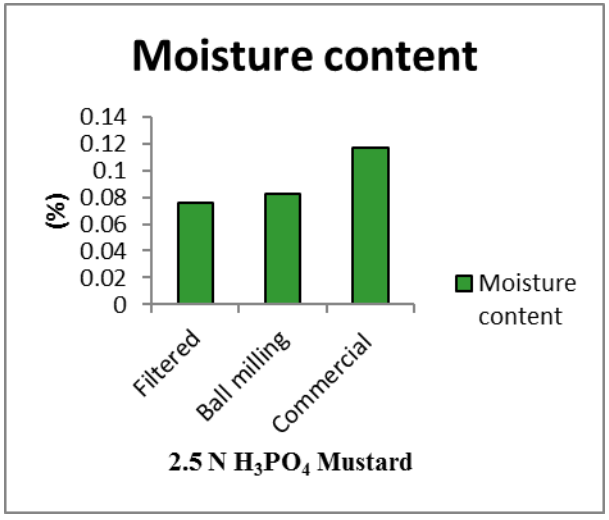


Figure 4.3 (d): Mean of moisture content

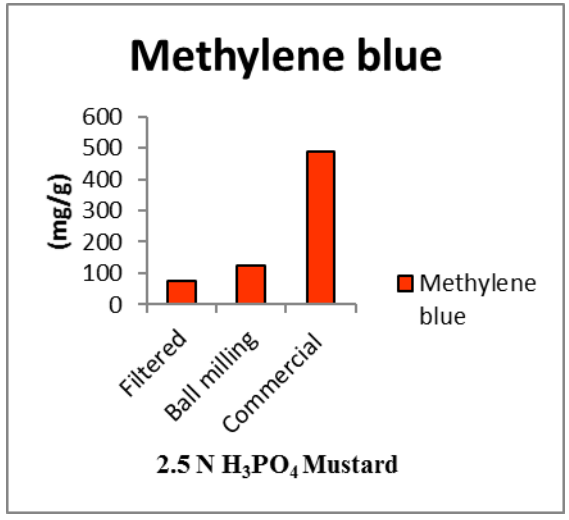


Figure 4.3 (e): Mean of methylene blue

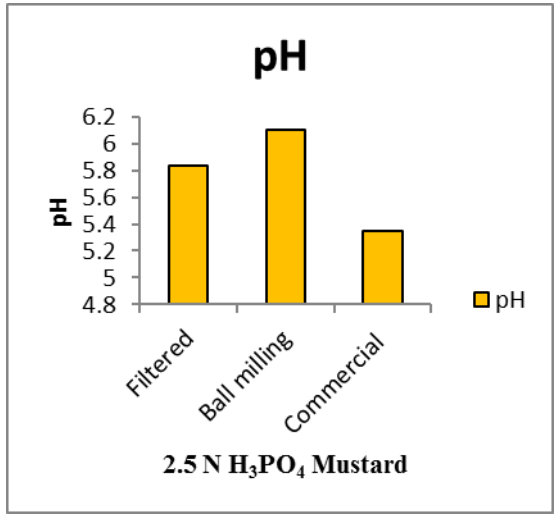


Figure 4.3 (f): Mean of ash pH

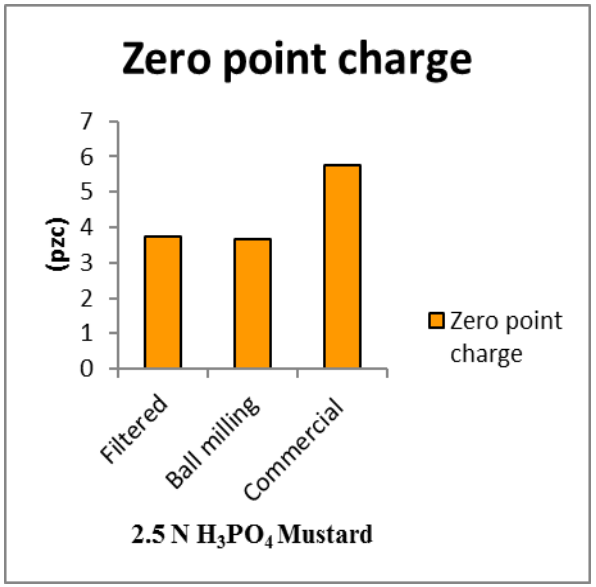


Figure 4.3 (g): Mean of zero point charge

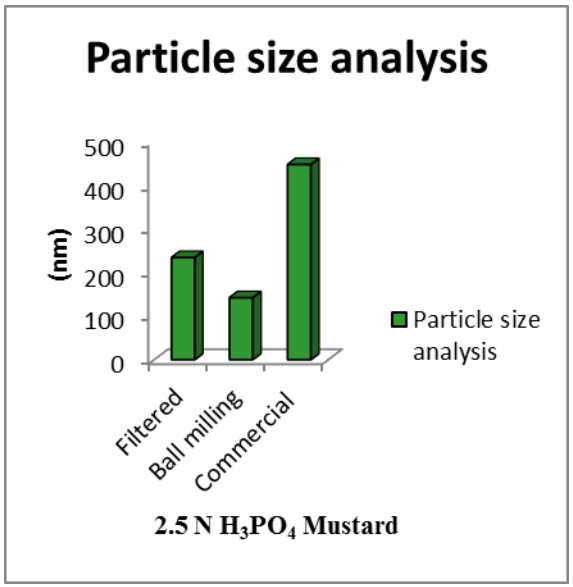


Figure 4.3 (h): Mean of particle size analysis

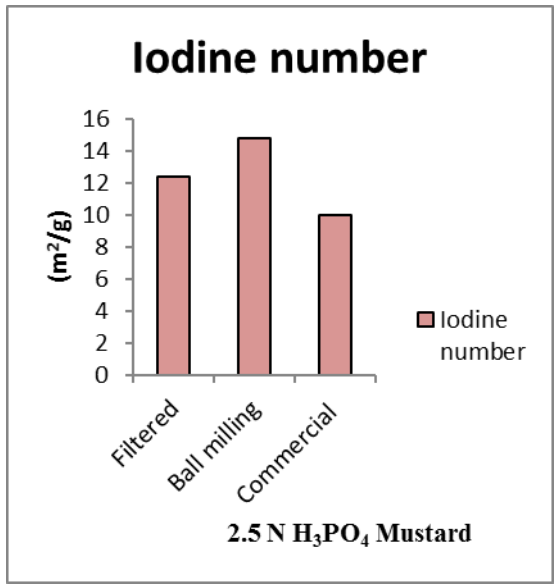


Figure 4.3 (i): Mean of ash iodine number

Figure 4.3: Characterizations of 2.5 N H_3PO_4 Mustard activated carbons

4.1.4 Characterization of activated carbon in metal complex dye using 2.5 N H_2SO_4 Almond activated carbon

Table 4.4: Characterization of 2.5 N H_2SO_4 Almond activated carbon

Activated carbons	Treatment	Bulk Density (g/cm^3)	Porosity (%)	Ash content (%)	Moisture content (%)	Methylene blue (mg/g)	pH	Zero point charge (pzc)	Particle size analysis (nm)	Iodine number (m^2/g)
2.5 N H_2SO_4 Almond	Filtered	4.700	3.600	0.127	0.064	107.000	7.420	6.713	1706.000	19.767
	Ball milling	5.233	3.633	0.172	0.071	116.000	6.973	6.460	825.000	21.5333
	Commercial activated carbon	6.467	6.533	0.118	0.117	488.000	5.343	5.750	447.667	10.000
	S. Ed(±)	**0.1610**	**0.1540**	**0.0253**	**0.0066**	**1.6330**	**0.0648**	**0.0949**	**1.2766**	**5.2674**
	$CD_{(0.05)}$ for activated carbon	**0.3940**	**0.3767**	**NS**	**0.0161**	**3.9958**	**0.1584**	**0.2322**	**3.1237**	**NS**

Foot note: - Data given are mean of three replications.

The table 4.4 and figure 4.4 (a-i), showed the characteristics of activated carbon 2.5 N H_2SO_4 of almond. The result registered that some of the characteristics of 2.5 N H_2SO_4 of almond activated carbon such as bulk density, porosity, ash content, moisture content, methylene blue, and iodine number showed a decreasing trend in case of filtered activated carbon. Besides, pH, zero point charge and particle size found to be decreased in ball milling activated carbon. From the statistical analysis, it was found that all the characters except iodine number and ash content found to be significant at 5% level with respect to 2.5 N H_2SO_4 of almond activated carbon.

Bulk density was recorded in commercial activated carbon (6.467g/cm^3), ball milling (5.233g/cm^3), and filtered (4.700g/cm^3) respectively. Porosity was obtained in commercial activated carbon (6.533%), ball milling (3.633%), and filtered (3.600%) respectively. Ash content found in commercial activated carbon was (0.118%), ball milling (0.172%), and filtered (0.127%) respectively. Moisture content exhibited in commercial activated carbons was (0.117%), ball milling (0.071%), and filtered (0.064%) respectively. The methylene blue in commercial activated carbons was obtained (488.000mg/g), ball milling (116.000mg/g), and filtered (107.000mg/g) respectively. The pH was recorded in commercial activated carbon as (5.343), ball milling (6.973), and filtered (7.420) respectively. Zero-point charges were obtained in commercial activated carbon (5.750pzc), ball milling (6.460pzc), and filtered (6.713pzc) respectively. Particle size analysis was recorded in commercial activated carbon (447.667nm), ball milling (825.000nm), and filtered (1706.000nm). The iodine numbers exhibited in commercial activated carbon were (10.000m^2/g), ball milling (21.5333m^2/g), and filtered (19.767m^2/g) respectively.

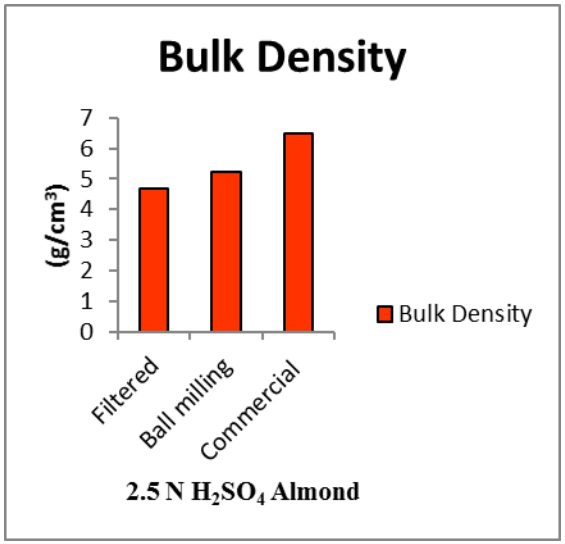


Figure 4.4 (a): Mean of bulk density

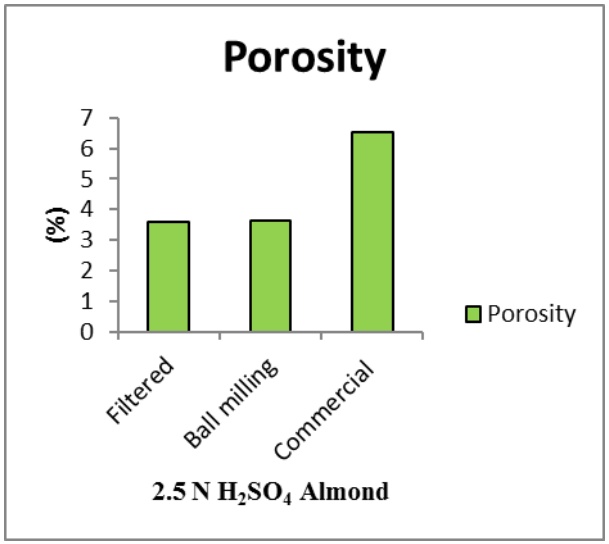


Figure 4.4 (b): Mean of Porosity

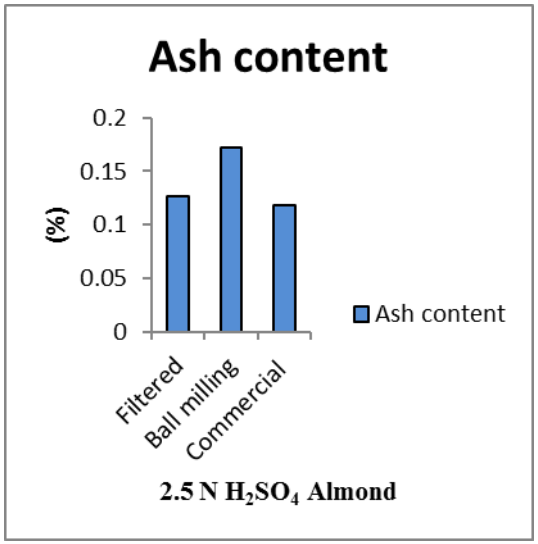


Figure 4.4 (c): Mean of ash content

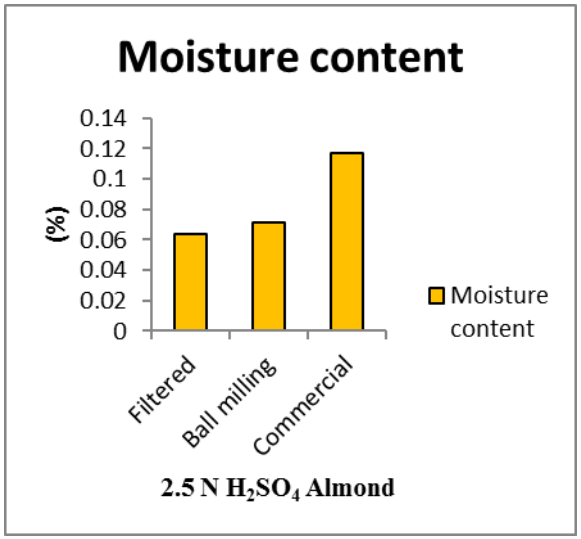


Figure 4.4 (d): Mean of moisture content

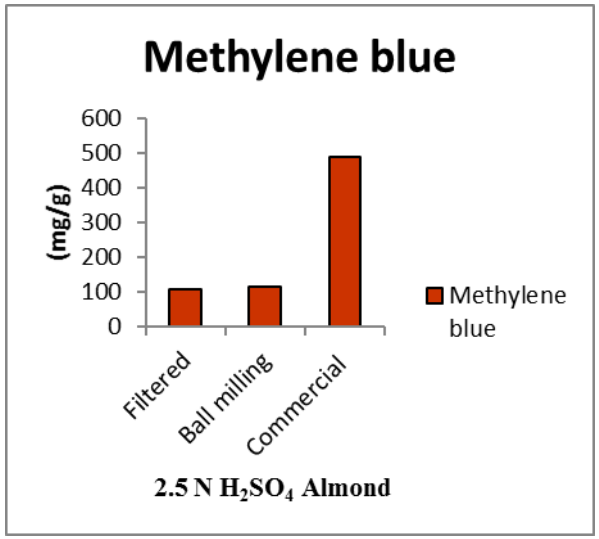


Figure 4.4 (e): Mean of methylene blue

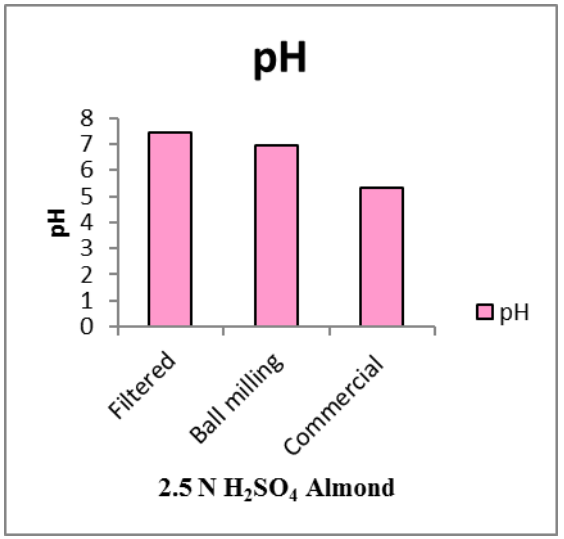
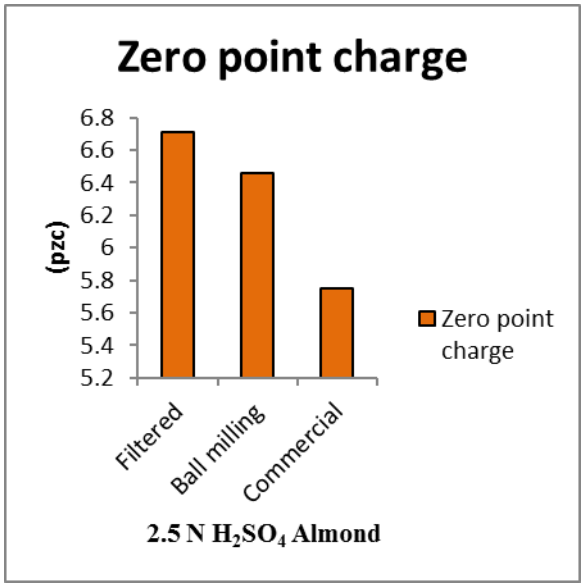
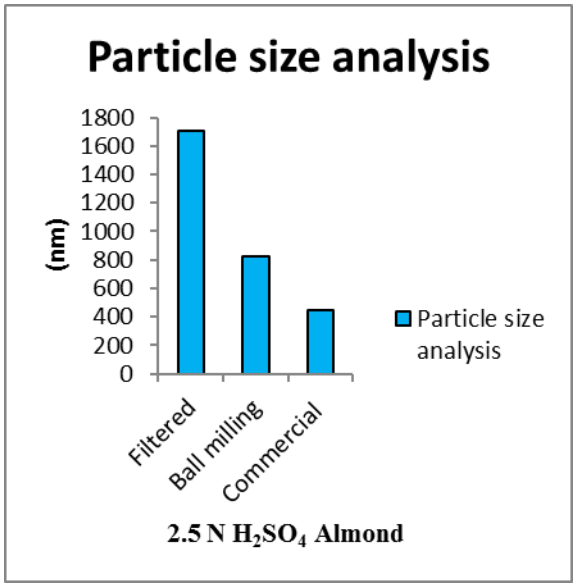


Figure 4.4 (f): Mean of ash pH





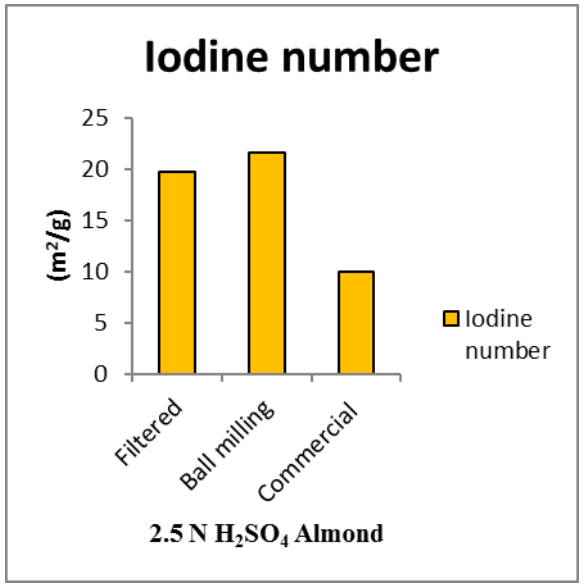


Figure 4.4 (g): Mean of zero point charge

Figure 4.4 (h): Mean of particle size analysis

Figure 4.4 (i): Mean of ash iodine number

Figure 4.4: Characterizations of 2.5 N H_2SO_4 Almond activated carbons

4.1.5 Characterization of activated carbon in metal complex dye using 2.5 N H_3PO_4 Rice Bran activated carbon

Table 4.5: Characterization of 2.5 N H_3PO_4 Rice Bran activated carbon

Activated carbons	Treatment	Bulk Density (g/cm^3)	Porosity (%)	Ash content (%)	Moisture content (%)	Methylene blue (mg/g)	pH	Zero point charge (pzc)	Particle size analysis (nm)	Iodine number (m^2/g)
2.5 N H_3PO_4 Rice Bran	Filtered	3.533	3.367	0.155	0.071	40.000	6.450	3.653	766.333	16.667
	Ball milling	4.400	3.467	0.160	0.056	56.000	6.997	3.913	390.333	16.100
	Commercial activated carbon	6.467	6.533	0.118	0.117	488.000	5.343	5.750	447.667	10.000
	S. Ed(±)	**0.1981**	**0.1700**	**0.0079**	**0.0065**	**1.6330**	**0.1561**	**0.1053**	**1.2472**	**2.4587**
	$CD_{(0.05)}$ for activated carbon	**0.4848**	**0.4159**	**0.0194**	**0.0160**	**3.9958**	**0.3820**	**0.2576**	**3.0518**	**NS**

Foot note: - Data given are mean of three replications.

The table 4.5 and figure 4.5 (a-i), depicted the characteristics of activated carbon 2.5 N H_3PO_4 of rice bran. The result showed that some of the characteristics of 2.5 N H_3PO_4 of rice bran activated carbon such as bulk density, porosity, ash content, methylene blue, pH and zero point charge showed a decreasing trend in case of filtered activated carbon. In addition, particle size, moisture content and iodine number found to be decreased in ball milling activated carbon. From the statistical analysis, it was found that all the characters except iodine number found to be significant at 5% level with respect to 2.5 N H_3PO_4 of rice bran activated carbon.

Bulk density was recorded in commercial activated carbon (6.467g/cm^3), ball milling (4.400g/cm^3), and filtered (3.533g/cm^3) respectively. Porosity was found in commercial activated carbon (6.533%), ball milling (3.467%), and filtered (3.367%) respectively. Ash content was obtained in commercial activated carbon (0.118%), ball milling (0.160%), and filtered (0.155%) respectively. The moisture content was exhibited in commercial activated carbon (0.117%), ball milling (0.056%), and filtered (0.071%) respectively. The methylene blue was found in commercial activated carbon (488.000mg/g), ball milling (56.000mg/g), and filtered (40.000mg/g) respectively. The pH was recorded in commercial activated carbon (5.343), ball milling (6.997), and filtered (6.450) respectively. The zero point charges were recorded at (5.750pzc), ball milling at (3.913pzc) and filtered at (3.653pzc) respectively. The particle size analysis was obtained in commercial activated carbon as (447.667nm), ball milling (390.333nm), and filtered (766.333nm) respectively. The iodine numbers were exhibited in commercial activated carbon (10.000m^2/g), ball milling (16.100m^2/g), and filtered (16.667m^2/g) respectively.

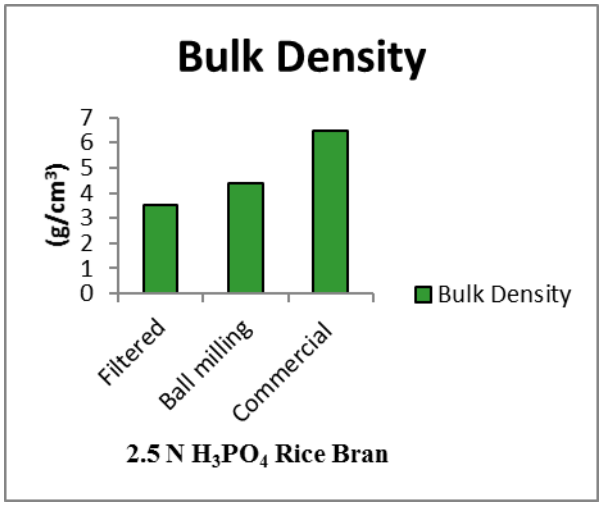


Figure 4.5 (a): Mean of bulk density

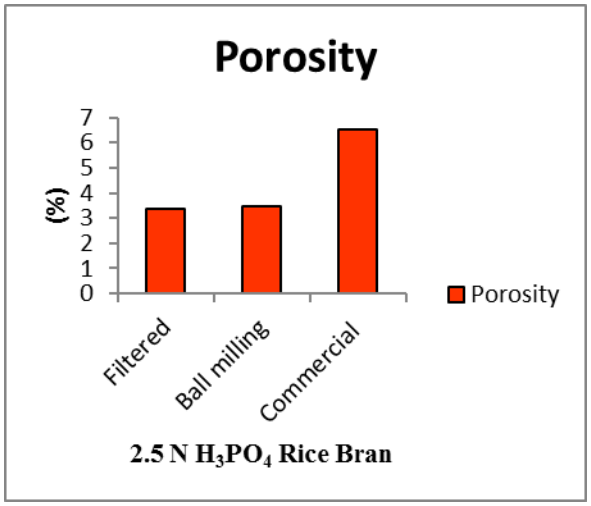


Figure 4.5 (b): Mean of Porosity

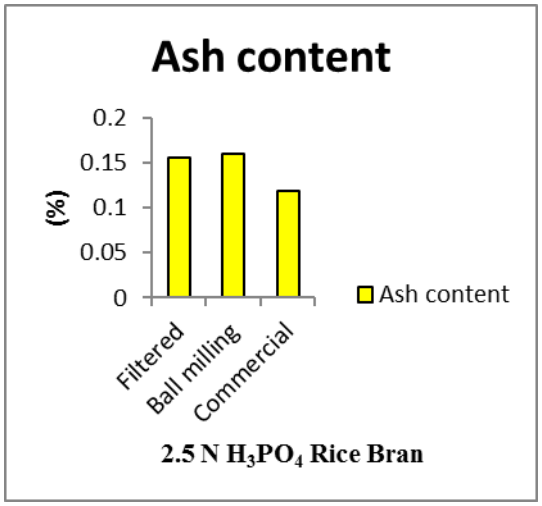


Figure 4.5 (c): Mean of ash content

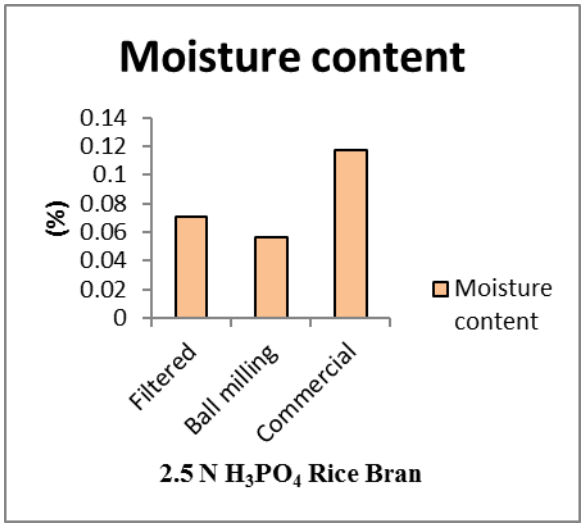


Figure 4.5 (d): Mean of moisture content

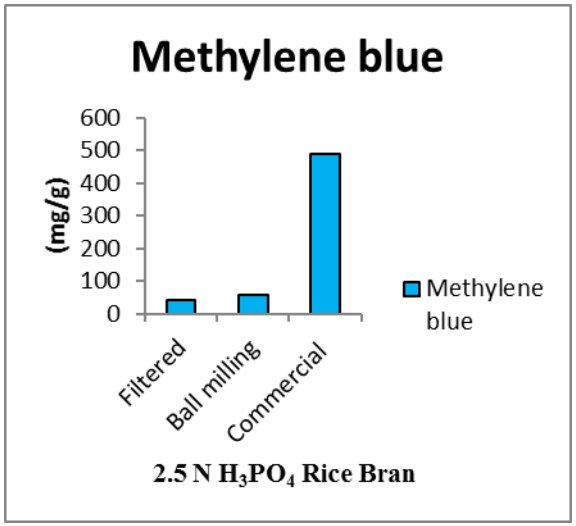


Figure 4.5 (e): Mean of methylene blue

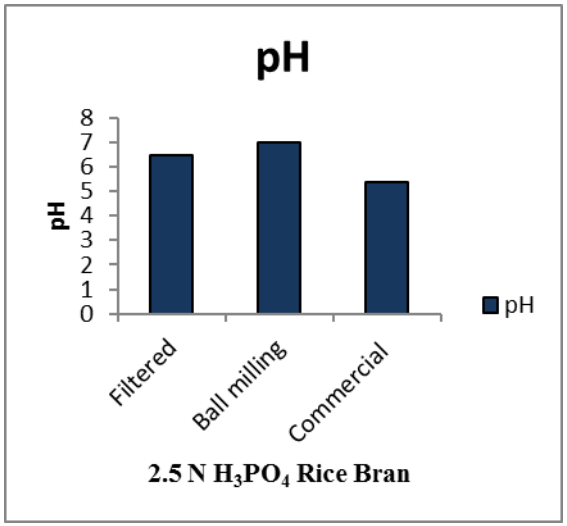


Figure 4.5 (f): Mean of ash pH

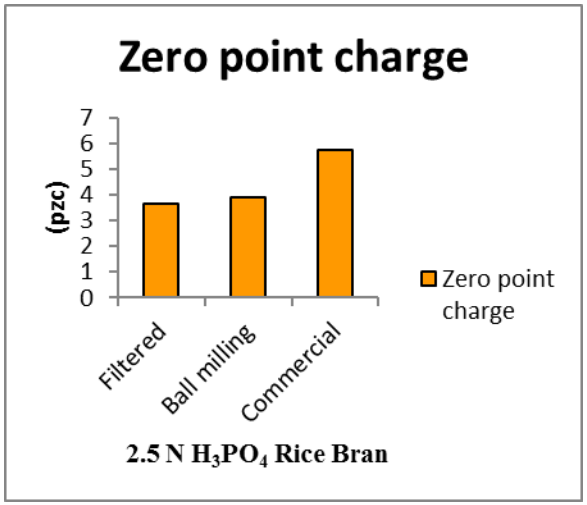


Figure 4.5 (g): Mean of zero point charge

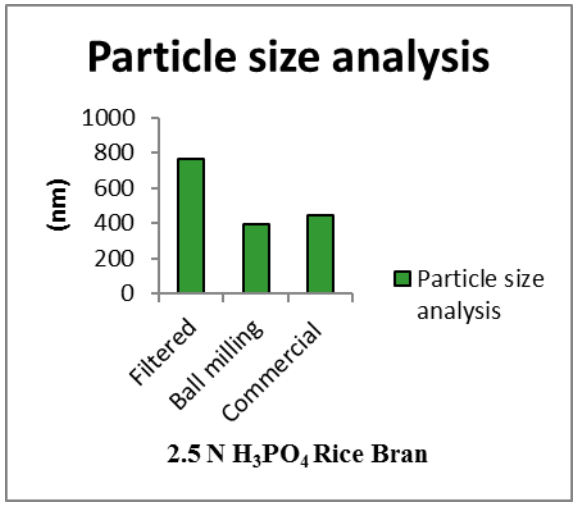


Figure 4.5 (h): Mean of particle size analysis

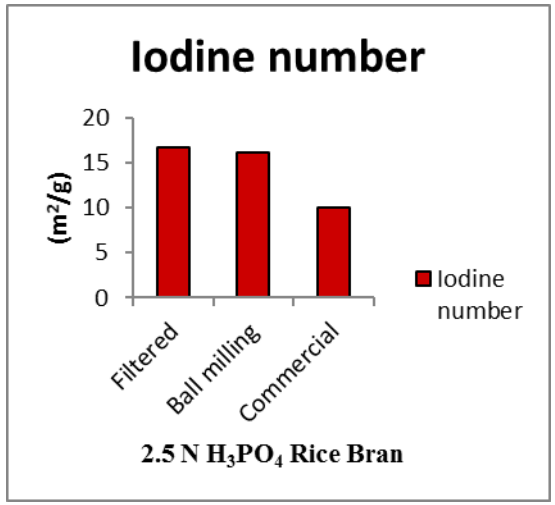


Figure 4.5 (i): Mean of ash iodine number

Figure 4.5: Characterizations of 2.5 N H_3PO_4 Rice Bran activated carbons

4.1.6 Characterization of activated carbon in metal complex dye using 2.5 N H_2SO_4 Sesame activated carbon

Table 4.6: Characterization of 2.5 N H_2SO_4 Sesame activated carbon

Activated carbons	Treatment	Bulk Density (g/cm^3)	Porosity (%)	Ash content (%)	Moisture content (%)	Methylene blue (mg/g)	pH	Zero point charge (pzc)	Particle size analysis (nm)	Iodine number (m^2/g)
2.5 N H_2SO_4 Sesame	Filtered	4.333	3.500	0.159	0.037	78.667	6.407	5.813	1382.333	16.667
	Ball milling	4.600	3.600	0.160	0.033	88.000	6.527	5.643	1326.667	16.400
	Commercial activated carbon	6.467	6.533	0.118	0.117	488.000	5.343	5.750	447.667	10.000
	S. Ed(±)	**0.1981**	**0.2177**	**0.0070**	**0.0012**	**1.7212**	**0.0653**	**0.1232**	**1.6997**	**2.5370**
	$CD_{(0.05)}$ for activated carbon	**0.4848**	**0.5328**	**0.0171**	**0.0028**	**4.2117**	**0.1598**	**NS**	**4.1590**	**NS**

Foot note: - Data given are mean of three replications.

The table 4.6 and figure 4.6 (a-i), represented the characteristics of activated carbon 2.5 N H_2SO_4 of sesame. The result showed that some of the characteristics of 2.5 N H_2SO_4 of sesame activated carbon such as bulk density, porosity, ash content, methylene blue and pH and showed decreasing trend in case of filtered activated carbon. In addition, particle size, zero point charge, moisture content and iodine number found to be decreased in ball milling activated carbon. From the statistical analysis, it was found that all the characters except iodine number and zero point charge found to be significant at 5% level with respect to 2.5 N H_2SO_4 of sesame activated carbon.

Bulk density was obtained in commercial activated carbon (6.467g/cm^3), ball milling (4.600g/cm^3), and filtered (4.333g/cm^3) respectively. Porosity was recorded in commercial activated carbon (6.533%), ball milling (3.600%), and filtered (3.500%) respectively. Ash contents were found in commercial activated carbon (0.118%), ball milling (0.160%), and filtered (0.159%) respectively. Moisture contents were exhibited in commercial activated carbon (0.117%), ball milling (0.033%), and filtered (0.037%) respectively. The methylene blue in commercial activated carbons was found (488.000mg/g), ball milling (88.000mg/g), and in filtered (78.667mg/g). The pH was exhibited in commercial activated carbon (5.343), ball milling (6.527), and filtered (6.407). The zero point charges were found in commercial activated carbon at (5.750pzc), ball milling at (5.643pzc), and filtered at (5.813pzc). The particle size analysis was recorded in commercial activated carbon (447.667nm), ball milling (1326.667nm), and filtered (1382.333nm) respectively. The iodine numbers were recorded in commercial activated carbon (10.000m^2/g), ball milling (16.400m^2/g), and filtered (16.667m^2/g) respectively.

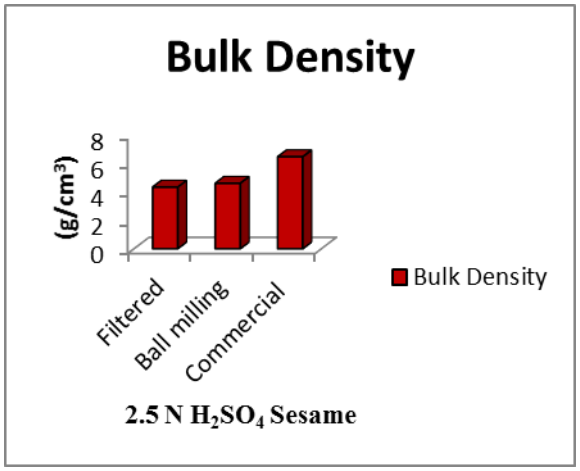


Figure 4.6 (a): Mean of bulk density

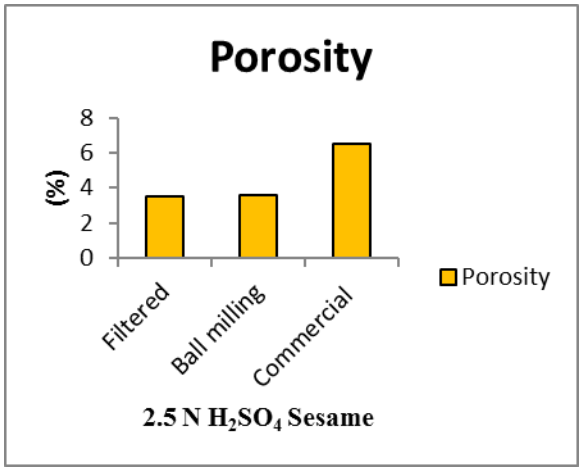


Figure 4.6 (b): Mean of Porosity

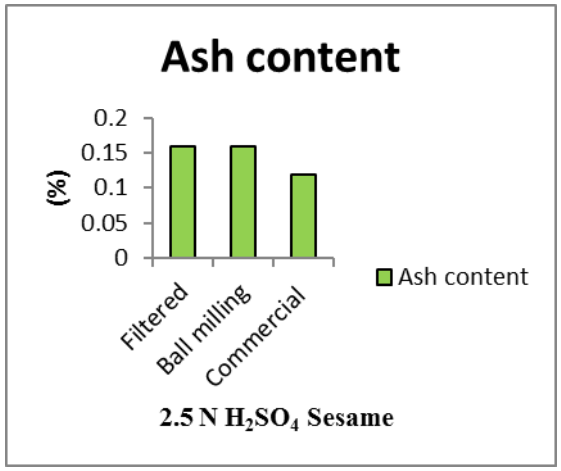


Figure 4.6 (c): Mean of ash content

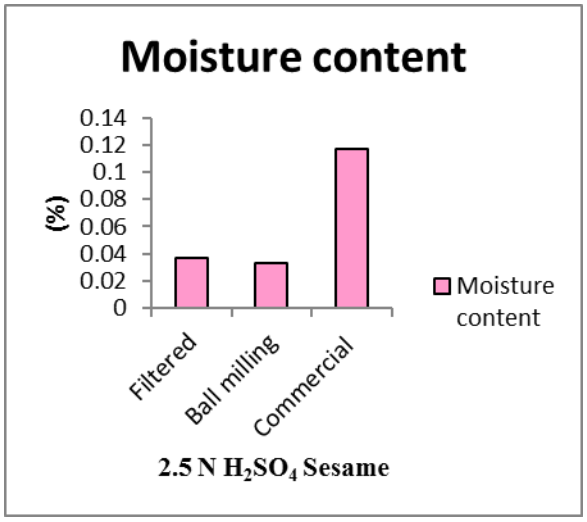


Figure 4.6 (d): Mean of moisture content

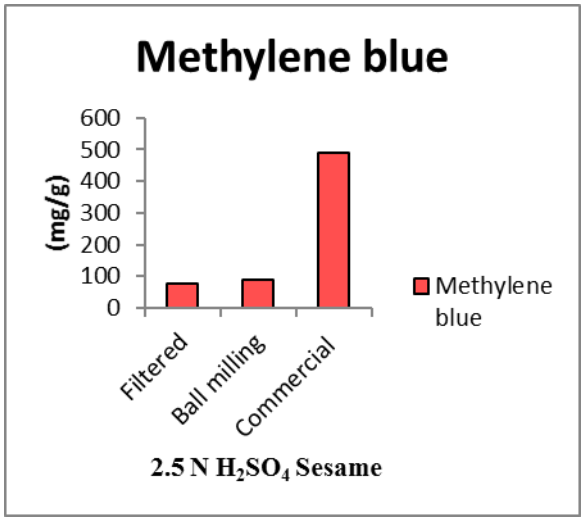


Figure 4.6 (e): Mean of methylene blue

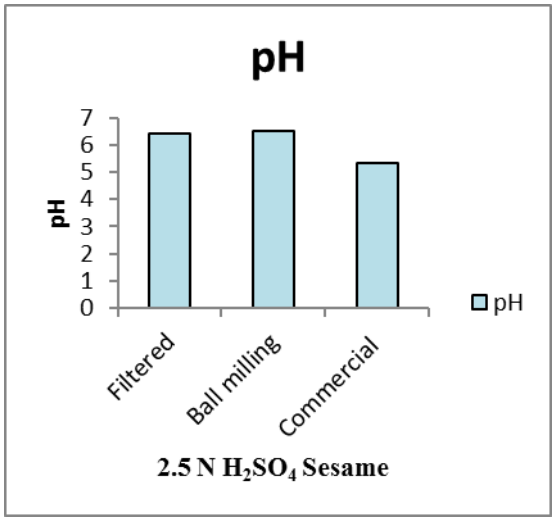


Figure 4.6 (f): Mean of ash pH

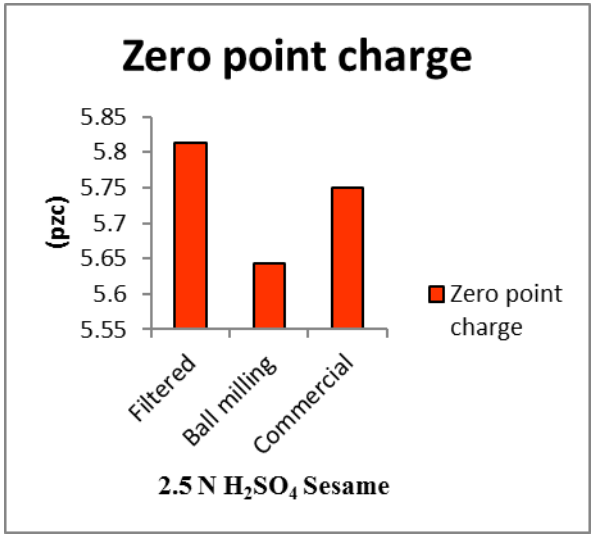


Figure 4.6 (g): Mean of zero point charge

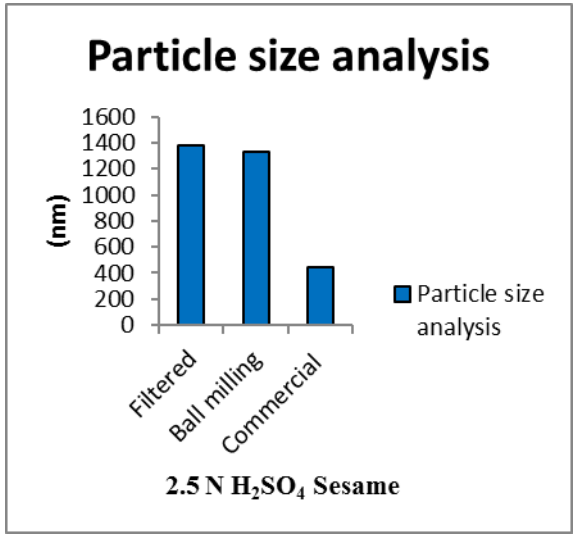


Figure 4.6 (h): Mean of particle size analysis

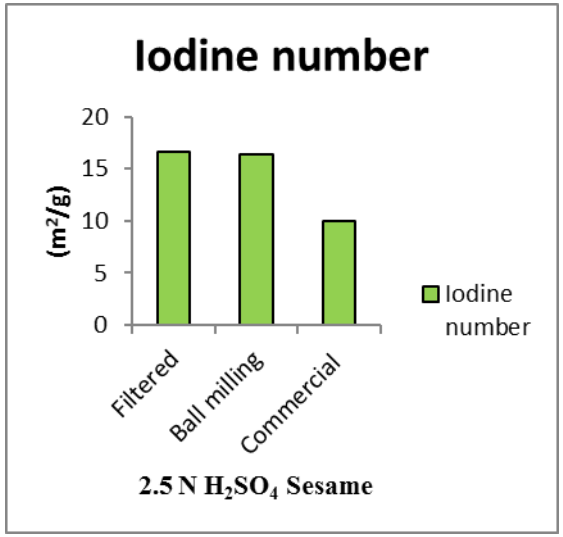


Figure 4.6 (i): Mean of ash iodine number

Figure 4.6: Characterizations of 2.5 N H_2SO_4 Sesame activated carbons

Ademiluyi and West (2012) studied the physio-chemical parameters like bulk density, methylene blue, iodine number and ash content of different chemical activations on the adsorption of heavy metals ions using activated carbons from waste materials such as bamboo, palm kernel shell, and coconut shell found that palm kernel shell had the highest bulk density and bamboo recorded least. The methylene blue adsorption capacity for bamboo was higher than that of coconut and palm kernel shell after activation, which means that bamboo has a higher mesopore structure than activated carbon from coconut shell and palm kernel shell. Comparing the iodine number of activated carbon from bamboo, coconut, and palm kernel shell, it was observed that the iodine number of bamboo activated carbon was higher than the activated carbon produced from coconut and palm kernel shells which might be due to the larger micropore structure from bamboo activated carbon. Higher ash content in activated carbon from palm kernel than bamboo and coconut reduced the overall activity of activated carbon from palm kernel.

Ekpete and Horsfall (2011) studied the characteristics of activated carbon's Point of Zero Charge **(**pHpzc) and porosity of fluted activated carbon (FAC) and commercial activated carbon (CAC). Porosity was higher in FAC than CAC, which shows the highest number of pores present in a sample. The Point of Zero Charge **(**pHpzc) of the adsorption capacity of the activated carbon and the pH of the FAC and CAC were 6.7 and 7.0 and are in the acceptable range for most applications.

Elango and Govindasamy (2018) studied the characterization of pH, and moisture content of activated carbon from temple waste flowers prepared by direct pyrolysis process, sodium sulphate (Na_2SO_4), and potassium hydroxide (KOH) process respectively, and found that the pH of the effluent was 7.96 which was alkaline and at high pH metals become insoluble therefore it could be hazardous to the environment. The activated carbon showed a low amount of moisture content which showed that the carbon had more surface area and therefore adsorption was high and the solubility of carbon might be low.

Islam *et al.,* (2012) carried out work on preparation and characterization of activated carbon from biodiesel by-products to study the particle size of the activated carbon found that small-sized particles showed better activation, and the char yield at

small particles was low. A decrease in particle size helps the volatile matter to escape easily from the particle, thus enhancing the pore development.

4.2 SEM (Scanning electron micrograph images) analysis of activated carbon

The Scanning electron micrographs of 2.5 N H_2SO_4 and 2.5 N H_3PO_4 of treated almonds were presented in figure 4.7 (a and b). The surface corrugations of micro and nano size activated carbon were visible in the images. Yue (1995) reported that H_3PO_4 and H_2SO_4 reacted with oil cake create pores due to the evaporation of volatile gases during the pyrolysis process. The pores produced by the 2.5 N H_3PO_4 treatments were found to be smaller in size as compared to 2.5 N H_2SO_4 treatments. It was confirmed that the number of pores per unit area produced by H_3PO_4 treatment was high as mentioned (Table 4.1). As the Porous structure is high it helps to decrease the methylene blue value and color removal efficiencies of the activated carbon.

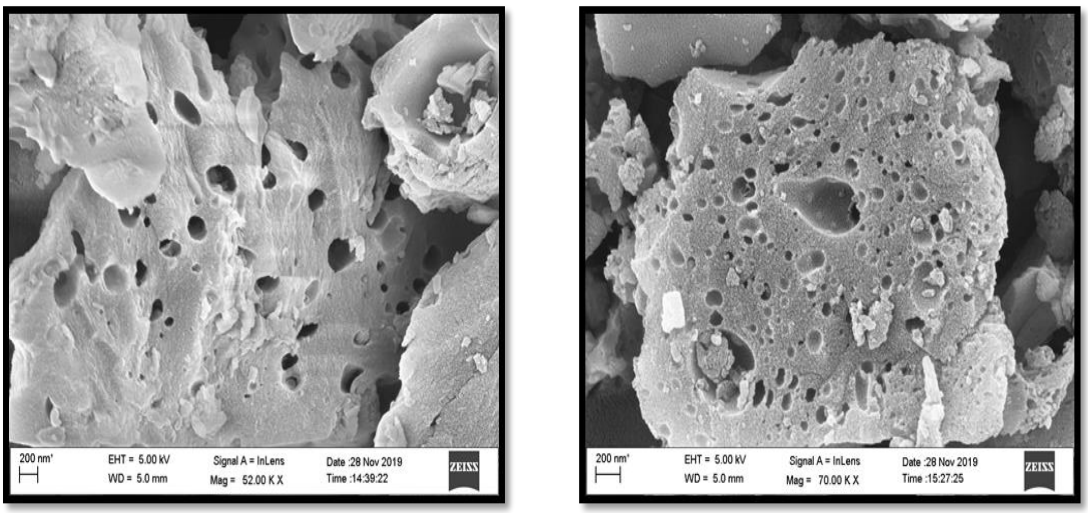


Figure 4.7: Scanning electron micrograph images of (a) 2.5 NH_2SO_4 treated and (b) 2.5 NH_3PO_4 treated almond

Mohan *et al.*, 2011 while working on the development of magnetic activated carbon that was prepared from almond shells for removal of trinitrophenol from water found that the SEM images of non-magnetic almond shells activated carbon (ASAC) and magnetic almond shells activated carbon (MASAC) showed porous surface with disorganized structural patterns that contain some original almond shell morphology. The macropore size distribution had discrete groups of pore sizes rather than a continuum, and micrographs showed a porous texture with small canals for ASAC and also showed some morphological changes due to iron oxide impregnation inside the pores of the carbon matrix.

4.3 Energy Dispersive X-Ray (EDX) analysis of activated carbon

Table 4.7: EDX analysis of almond activated carbon treated with 2.5 N H_3PO_4 and 2.5 N H_2SO_4

Activated carbon from	Elements (%)									
	C	O	Mg	Si	P	Ca	Na	K	S	Total
2.5 N H_3PO_4 treated almond	83.67	14.85	0.11	0.13	1.16	0.08	-	-	-	100
2.5 N H_2SO_4 treated almond	82.94	15.98	-	-	-	-	0.14	0.05	0.89	100

The EDX analysis of activated carbon prepared from the chemically treated 2.5 N H_3PO_4 and 2.5 N H_2SO_4 almond activated carbons were shown in table 4.7 and figure 4.8 (a and b). The table indicated that there were no significant changes in the carbon content of the activated carbons prepared by the activation of mineral acids, however; the carbon content was seemed to be slightly higher in phosphoric acid treated 2.5 N H_2SO_4 almond. A negligible quantity of Na, K, S and Mg, Si, P, C elements were observed in case of 2.5 N H_3PO_4 and 2.5 N H_2SO_4 almonds activated carbons respectively.

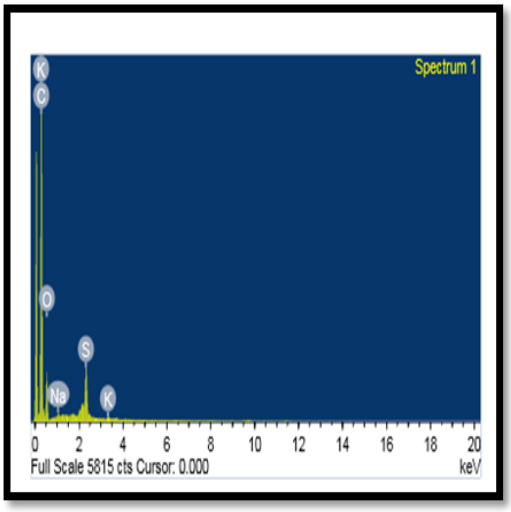
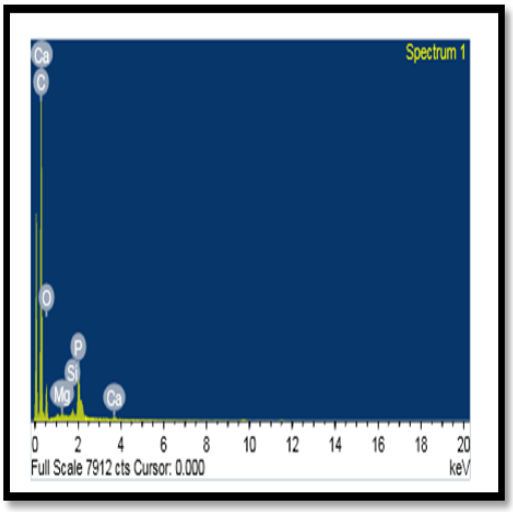


Figure 4.8: EDX images of (a) 2.5 N H_2SO_4 almond activated carbon



(b) 2.5 N H_3PO_4 almond activated carbon

Mohan *et al.*, 2011 while working on the development of magnetic activated carbon that was prepared from almond shells for removal of trinitrophenol from water found that the EDX analysis, spectra of non-magnetic activated carbon (ASAC) and magnetic activated carbon (MASAC) and found that their elemental composition where, ASAC contains carbon, oxygen, aluminum and no iron was observed in ASAC. MASAC contains iron and the aluminum peak present in ASAC disappeared in the MASAC.

4.4 Chemical Oxygen Demand

Table 4.8: COD of acid dye and metal complex dye effluent treated with 2.5 N H_2SO_4 and 2.5 N H_3PO_4 almond activated carbon

Dye effluent	**Activated carbon**	**Found**
Acid dye	2.5 N H_2SO_4 almond	5712 mg/l
	2.5 N H_3PO_4 almond	5605.60 mg/l
Metal complex dye	2.5 N H_2SO_4 almond	4896mg/l
	2.5 N H_3PO_4 almond	4804.80 mg/l

Table 4.8 depicted that the activated carbon 2.5 N H_2SO_4 and 2.5 N H_3PO_4 almonds treated with acid dye recorded COD value (5712mg/l), and (5605.60mg/l) respectively. Moreover, COD value of treated with metal complex dye effluent were found to be (4896mg/l) for 2.5 N H_2SO_4 and (4804.80mg/l) for 2.5 N H_3PO_4 almonds activated carbon respectively. A positive result was obtained in both the acid and metal complex dyes treated with 2.5 N H_3PO_4 almond than 2.5 N H_2SO_4 almond activated carbon.

Elango and Govindasamy (2018) have worked on removal of color from textile dyeing effluent using temple waste flowers as ecofriendly activated carbon to analyze the chemical oxygen demand (COD) opined that, the COD of the effluent was 2180 ppm, which was high compared to BIS indicated that there was high level of industrial wastes such as detergents, softeners, non biodegradable dyeing chemicals, formaldehyde based dye fixing agents etc. Present in the effluent thus incompatible for the survival of water living organisms.

4.5 Biological Oxygen Demand

Table 4.9: BOD of acid dye and metal complex dye effluent treated with 2.5 N H_2SO_4 and 2.5 N H_3PO_4 almond activated carbon

Dye effluent	Activated carbon	Found
Acid dye	2.5 N H_2SO_4 almond	1483.63 mg/l
	2.5 N H_3PO_4 almond	1456 mg/l
Metal complex dye	2.5 N H_2SO_4 almond	1271.68 mg/l
	2.5 N H_3PO_4 almond	1248.0 mg/l

Table 4.9 exhibited a positive trend that the activated carbon 2.5 N H_2SO_4 and 2.5 N H_3PO_4 almonds treated with acid dye recorded BOD as (1483.63mg/l) and (1456mg/l), respectively. In case of metal complex dye effluent, the BOD values were found as (1271.68mg/l) for 2.5 N H_2SO_4 and (1248.0mg/l) for 2.5 N H_3PO_4 almonds, respectively. Satisfactory results were obtained in both the acid dye and metal complex dye treated with 2.5 N H_3PO_4 than 2.5 N H_2SO_4 almond activated carbon.

Elango and Govindasamy (2018) while working on removal of color from textile dyeing effluent using temple waste flowers as environment friendly activated carbon to analyze the biological oxygen demand (BOD) opined that the BOD of the effluent was 760 ppm, which was higher compared to BIS limits indicated that there was very low amount of oxygen for living organisms for utilizing organic matter.

4.6 X-**Ray Diffraction (XRD) analysis of activated carbon: -** The X-ray diffraction patterns of the 2.5 N H_3PO_4 and 2.5 N H_2SO_4 almonds have been displayed in Figure 4.9 (a and b). The broad peak of the pattern revealed the presence of amorphous structure of the activated carbon with low crystallinity. In 2.5 N H_3PO_4 almond the peak at 29˚ might be associated with the presence of amorphous carbons with the diffraction pattern of (002) indicated the spacing of aromatic ring layer (Girgis, 2007). In 2.5 N H_2SO_4 almond the peak at 25˚ might be associated with the presence of amorphous carbons with the diffraction pattern of (002) indicated the spacing of aromatic ring layer (Manoj and Kunjomana, 2012).

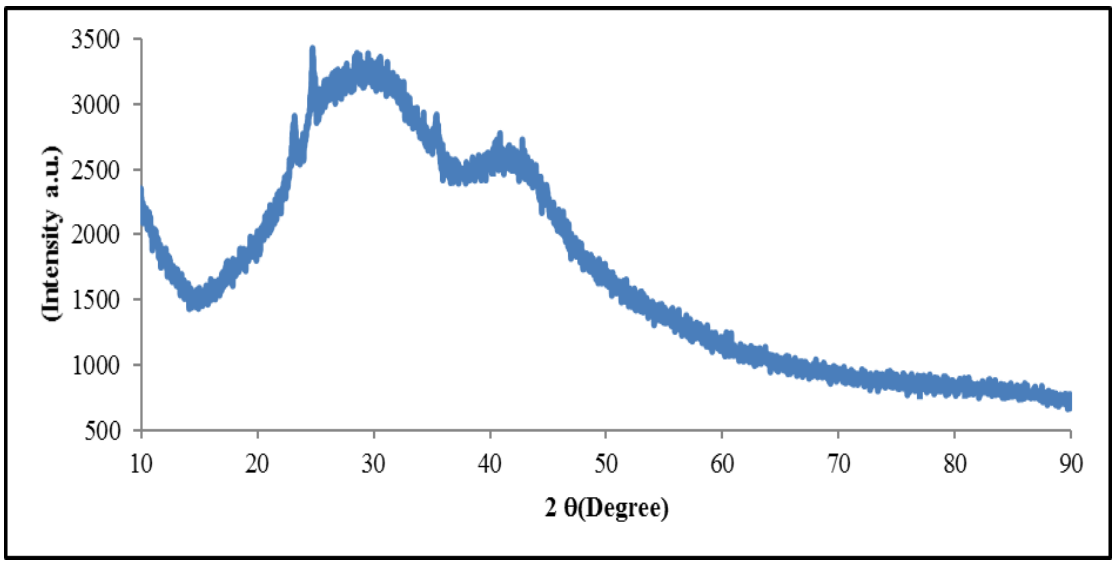


Figure 4.9 (a): XRD analysis of 2.5 N H_3PO_4 Almond activated carbon

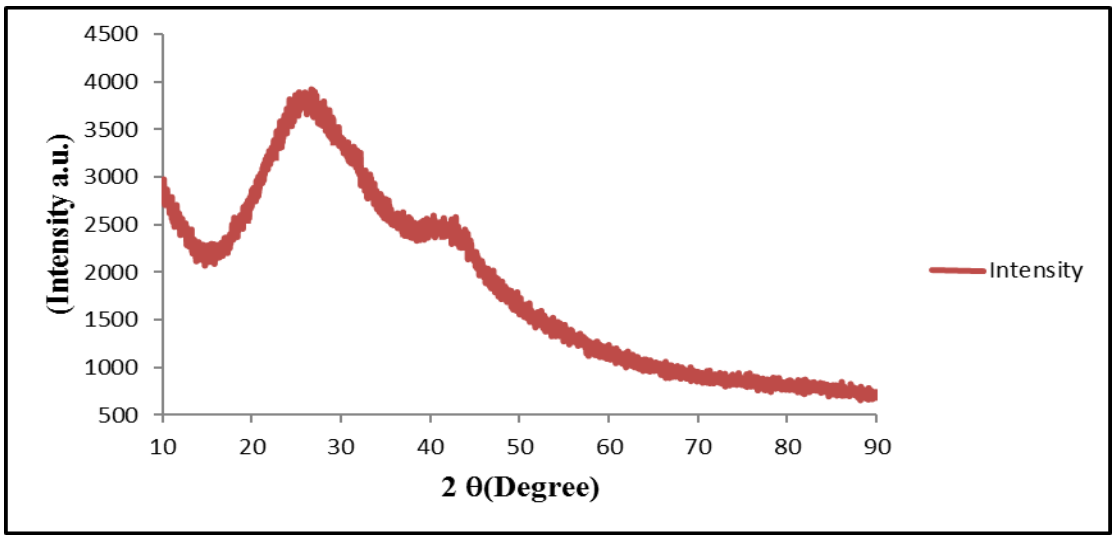


Figure 4.9 (b):- XRD analysis 2.5 N H_2SO_4 Almond activated carbon

4.7 Brunauer Emmet Teller (BET) analysis of activated carbon: - The BET analyses of 2.5 N H_3PO_4 and 2.5 N H_2SO_4 almonds were performed and the results were illustrated in Figure 4.10 (a and b) that there was strong association existed between the carbon content of the activated carbon and BET surface area. Pyrolysis lead to the degradation of organic material of oil cakes and enhanced the surface areas by 85.78m^2/g. Similar findings were given by Thangalazhy-Gopakumar *et al.*, (2015). In 2.5 N H_3PO_4 almond the surface area of the activated carbon was recorded as 16.14m^2/g. The pore radius and pore volume was recorded as 0.85nm and 0.029cc/g. In 2.5 N H_2SO_4 almond the surface area of the activated carbon was recorded as 64.28m^2/g. The pore radius and pore volume was recorded as 0.72nm and 0.040cc/g.

The specific surface area, pore volume and pore radius of the activated carbon were highly depended on the raw material, activating chemical and pyrolysis temperature which might be due to the difference in the decomposition of organic matters (Tomczyk *et al.*, 2020).

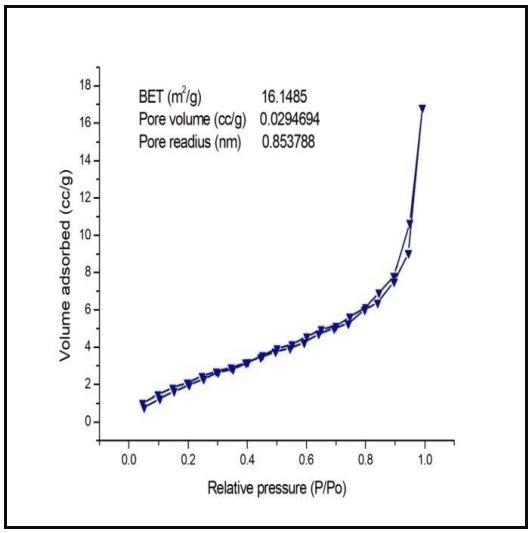


Figure 4.10 (a): BET analysis 2.5 N H_3PO_4 Almond

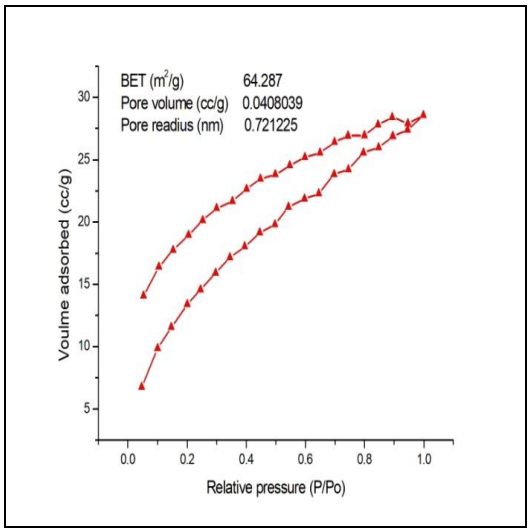


Figure 4.10 (b): BET analysis 2.5 N H_2SO_4 Almond

4.8 FTIR analysis of activated carbon

4.8.1 FTIR of 2.5 N H_3PO_4 Almond activated carbon

The FTIR result indicates activated carbon analysis of structural and functional groups. Most of the analysis of functional group were of the combination of filtered and ball milling activated carbon process which undergoes through the contacting process with simulation waste containing Pb^{2+} ions. The FTIR spectra of 2.5 N H_3PO_4 almond (filtered) activated carbon was given in figure 4.11(a). The graph of 2.5 N H_3PO_4 almond filtered displayed absorption bands at 1566cm^{-1} and 1277cm^{-1} assigned O–H, C, O, C-C and C–O bonds stretching modes, respectively. The broad band centered at1584 cm^{-1} indicated that a variety of C = C bonds exist besides those aromatic rings (Mohan, 2011). OMRI (2014) reported that the peaks at 1130cm^{-1}, were due to the vibrations of C–O stretching.

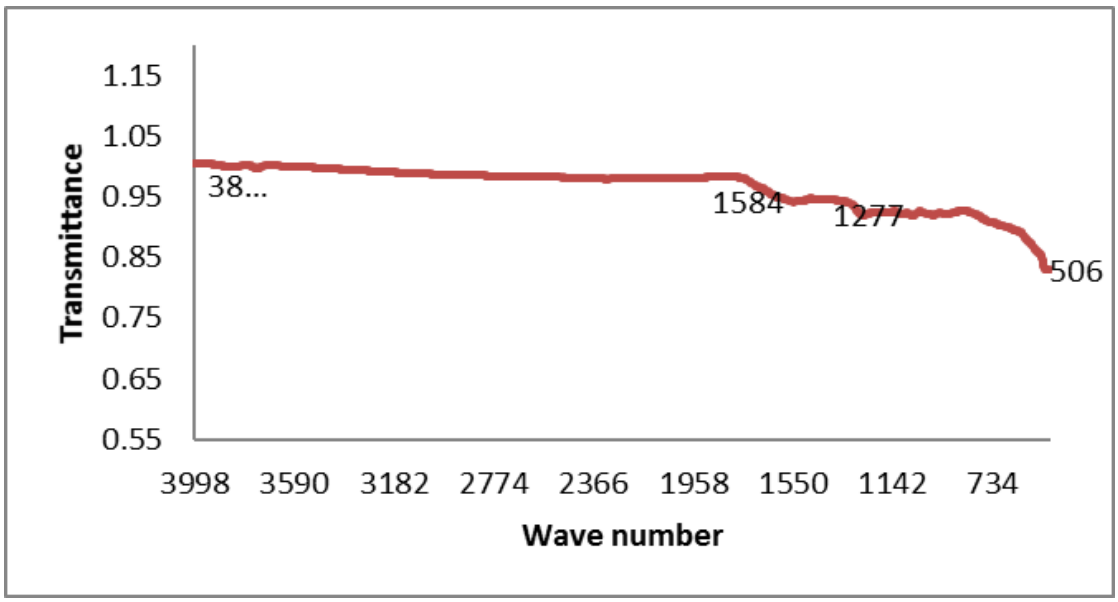


Figure 4.11 (a): FTIR spectra of 2.5 N H_3PO_4 Almond Filtered

FTIR analysis of 2.5 N H_3PO_4 Almond (ball milling) was shown in Figure 4.11 (b). Yakout and ElDeen (2016) reported that the FTIR spectra 1270 cm^{-1} was due to Phosphorous –Oxygen - Carbon stretch. Mansor *et al.*, (2012) reported that FTIR spectra 1142 cm^{-1} and 3736cm^{-1} was due to sulphonic acid group and hydroxyl group respectively.

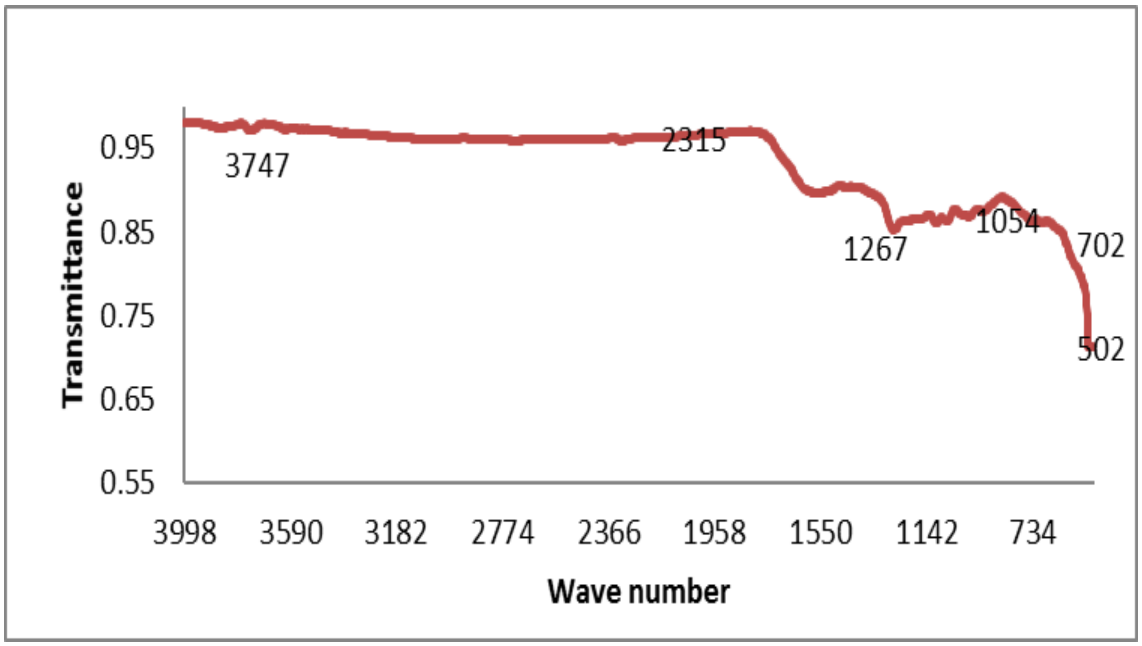


Figure 4.11 (b): FTIR spectra of 2.5 N H_3PO_4 Almond ball milling

4.8.2 FTIR of 1 N H_3PO_4 Coconut activated carbon

FTIR analysis depicted in figure 4.12 (a and b) resemblance in groups like C=O carbonyl, C=C aromatic and C-H aldehyde. In 1 N H_3PO_4 coconut (filtered)

$ZnCl_2$ and Na_2CO_3, the carbonyl group (C = O) was observed at 1740- 1700 cm^{-1}. A peak ranging from 1200-1000 cm-1 was associated with (C-O) stretch and 830cm^{-1} (Si-O) indicated the presence of silica (IkhtiarBakti, 2018). In 1 N H_3PO_4 coconut (ball milling) **t**he FTIR spectra at 1820- 1600 cm^{-1} associated with C=O carbonyl group. In the activated carbon sample the FTIR spectra appeared at 1701.29 cm^{-1} before activation and the activated carbon after absorption activation appeared in spectra 1698.40 cm^{-1}. The FTIR spectra appeared at 1694.54 cm^{-1} on the activated carbon after absorption (Saputro, 2019).

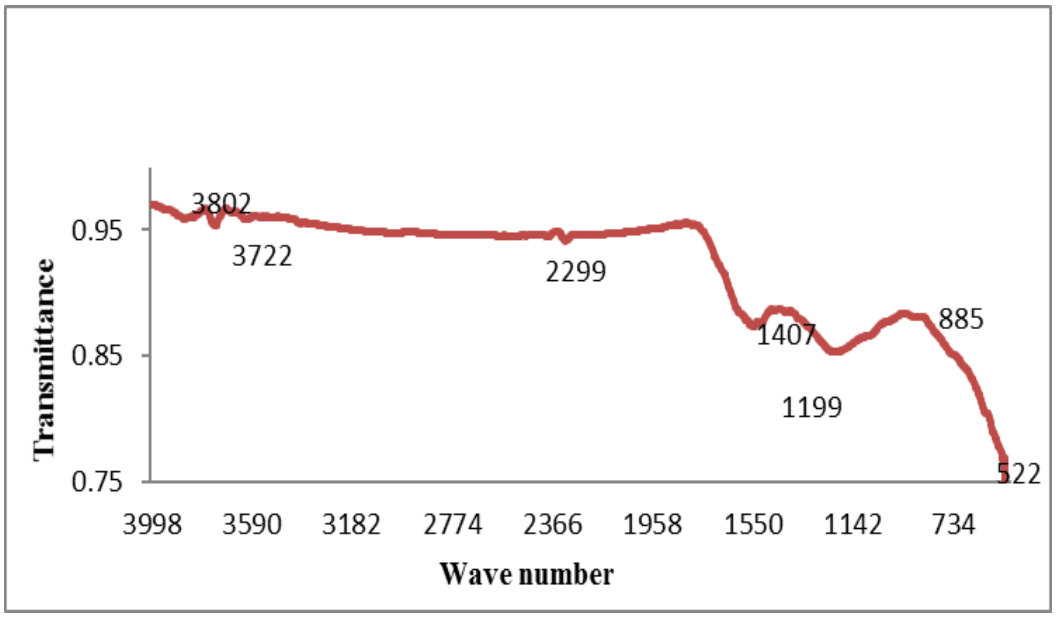


Figure 4.12 (a): FTIR spectra of 1 N H_3PO_4 Coconut (Filtered)

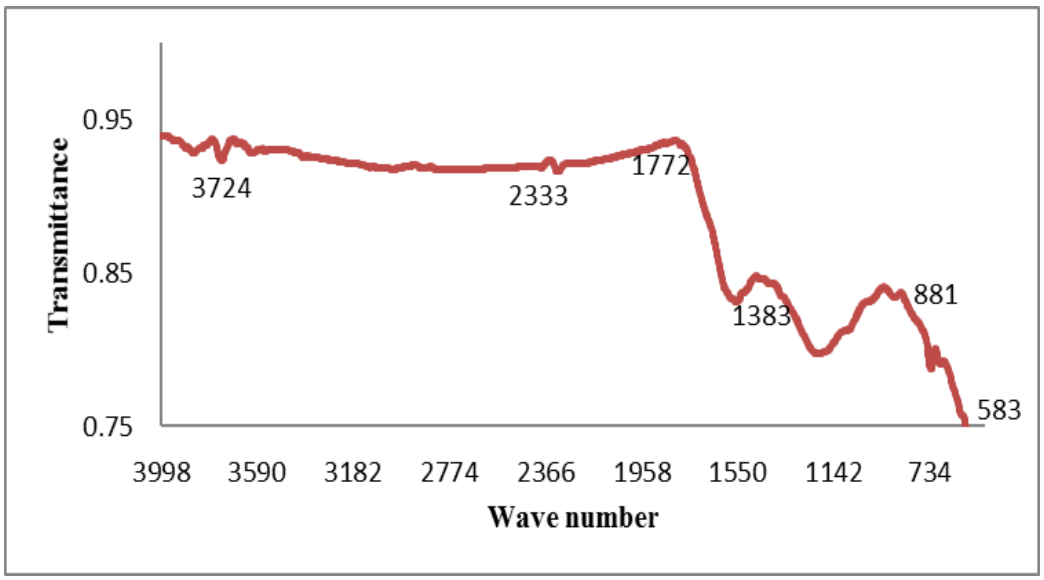


Figure 4.12 (b): FTIR spectra of 1 N H_3PO_4 Coconut (Ball milling)

4.8.3 FTIR of 2.5 N H_3PO_4 Mustard activated carbon

The FTIR analysis showed in figure 4.13 (a and b). The absorption bands observed in 2.5 N H_3PO_4 mustard (filtered) found to be in the range of 3464–3723 cm^{-1} are due to the chemisorbed water and surface hydroxyl groups. Some of the others bands originating from the sample are characteristics of –OH stretching 3454 cm^{-1} (Kaman Singh, 2012). In 2.5 N H_3PO_4 mustard (ball milling) the peaks in the range of 1630-1600 cm^{-1} showed the presence of H-O-H bonding and N-H bending vibrations of primary amines was observed in the peak 1628 cm^{-1}. 1072 cm^{-1} and 1040 cm^{-1} wave number was associated with the C-O stretching (Katiyar, 2015).

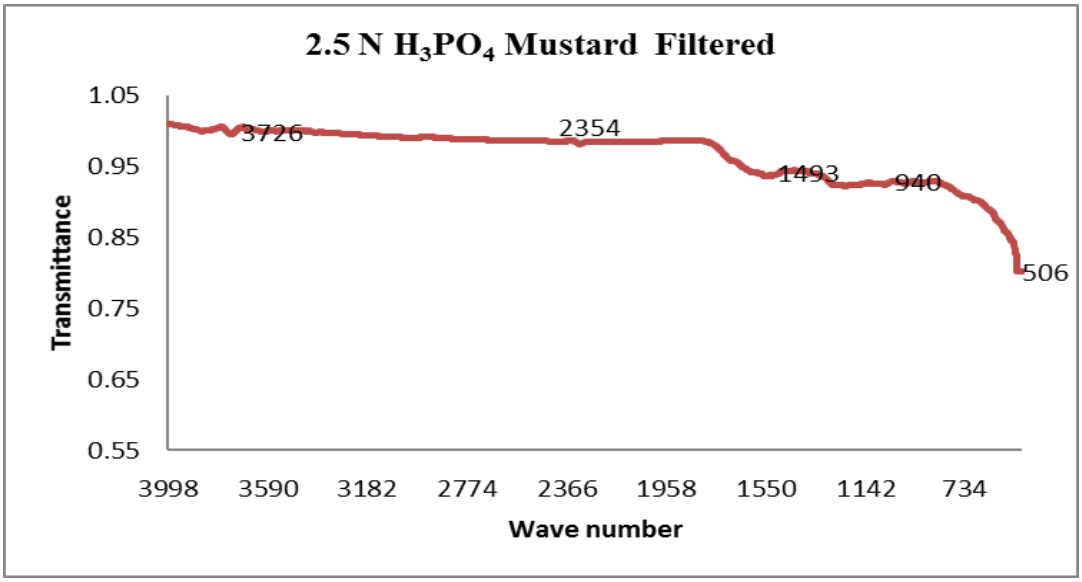


Figure 4.13 (a): FTIR spectra of 2.5 N H_3PO_4 Mustard Filtered

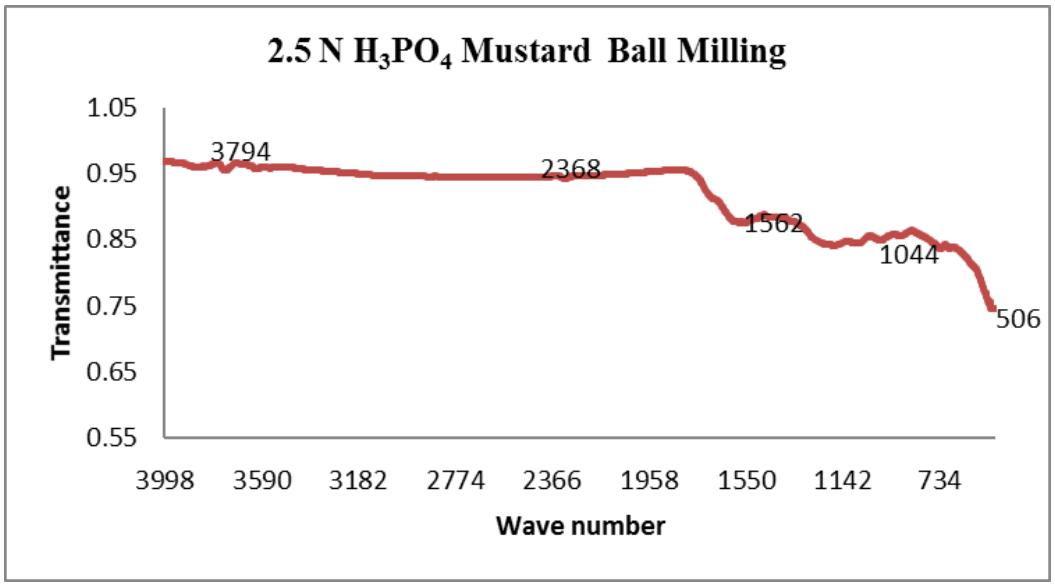


Figure 4.13 (b): FTIR spectra of 2.5 N H_3PO_4 Mustard ball milling

4.8.4 FTIR of 2.5 N H_2SO_4 Almond activated carbon

In 2.5 N H_2SO_4 almond (filtered) in figure 4.14 (a) showed that a strong conjugated C=C peak was observed around 1668 cm^{-1} and at 1191 cm^{-1} represented the stretching of C – O functional group. In 2.5 N H_2SO_4 almond (ball milling) in figure 4.14 (b) the FTIR spectral peak at 1589 cm^{-1} was due to the presence of carboxyl group and at 2096 cm^{-1} and 1793 cm^{-1} indicated the possible association of alkyne and ester groups. The peaks in the region 1142 cm^{-1} associated with C-Cl, C-F and C-O functional groups on the activated carbon surface (Hassan and Ahmed, 2011).

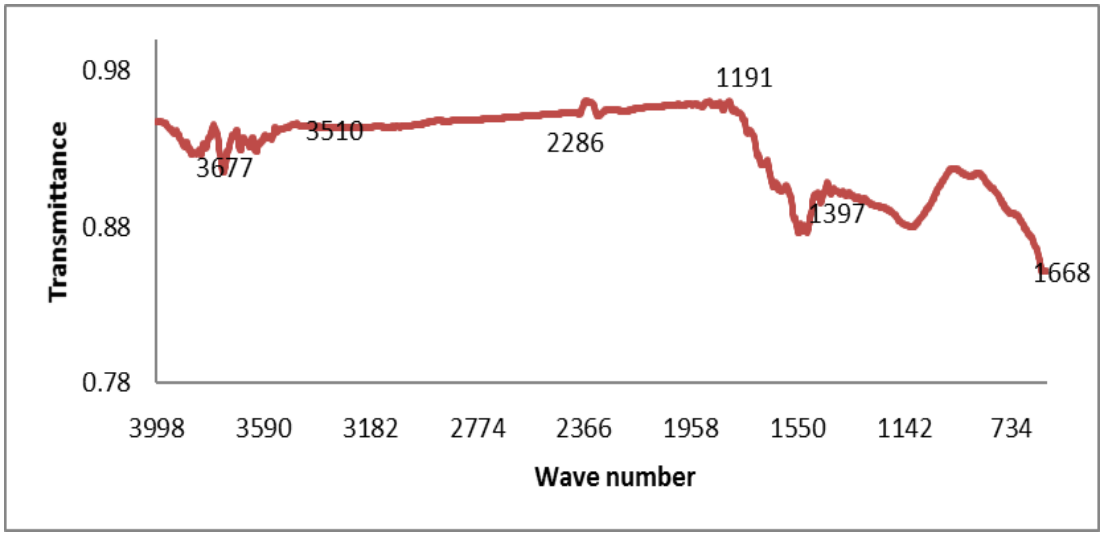


Figure 4.14 (a): FTIR spectra of 2.5 N H_2SO_4 Almond Filtered

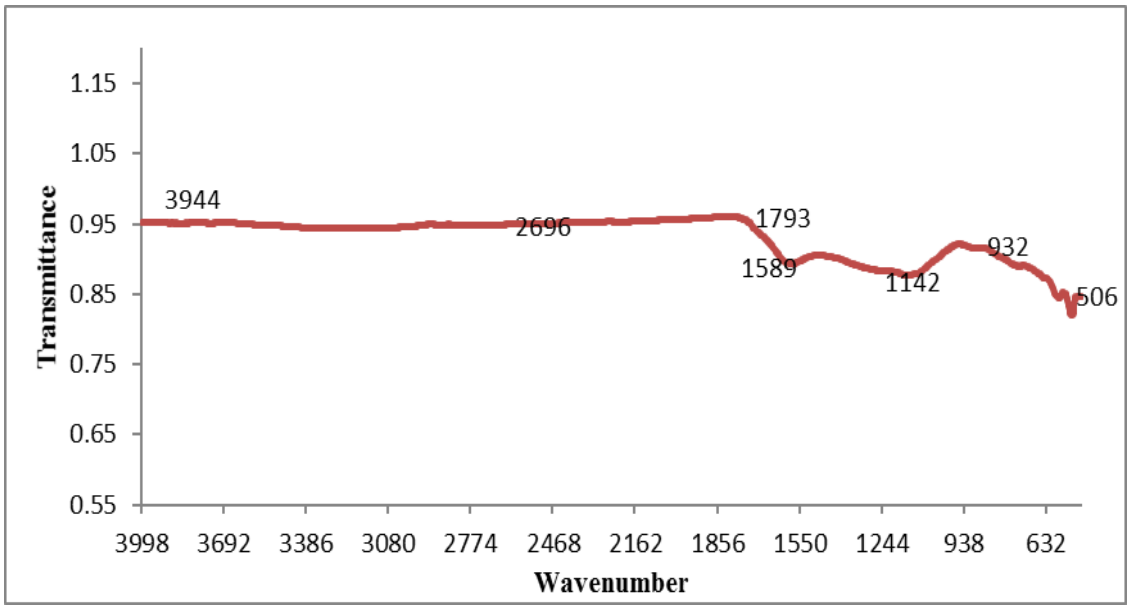


Figure 4.14 (b): FTIR spectra of 2.5 N H_2SO_4 Almond ball milling

4.8.5 FTIR of 2.5 N H_3PO_4 Rice Bran activated carbon

The FTIR spectrum showed in figure 4.15 (a and b). In 2.5 N H_3PO_4 rice bran (filtered) spectrum at 1300-900cm^{-1} could be caused by phosphorous-oxy-containing functional groups. The appearance of the broad band in the region 720-590 cm^{-1} assigned O-H group (Mohammad, 2015). In 2.5 N H_3PO_4 rice bran (ball milling) the presence of group C-O-H was indicated by absorption peak at 873 cm^{-1} (Chafidz *et al.*, 2018).

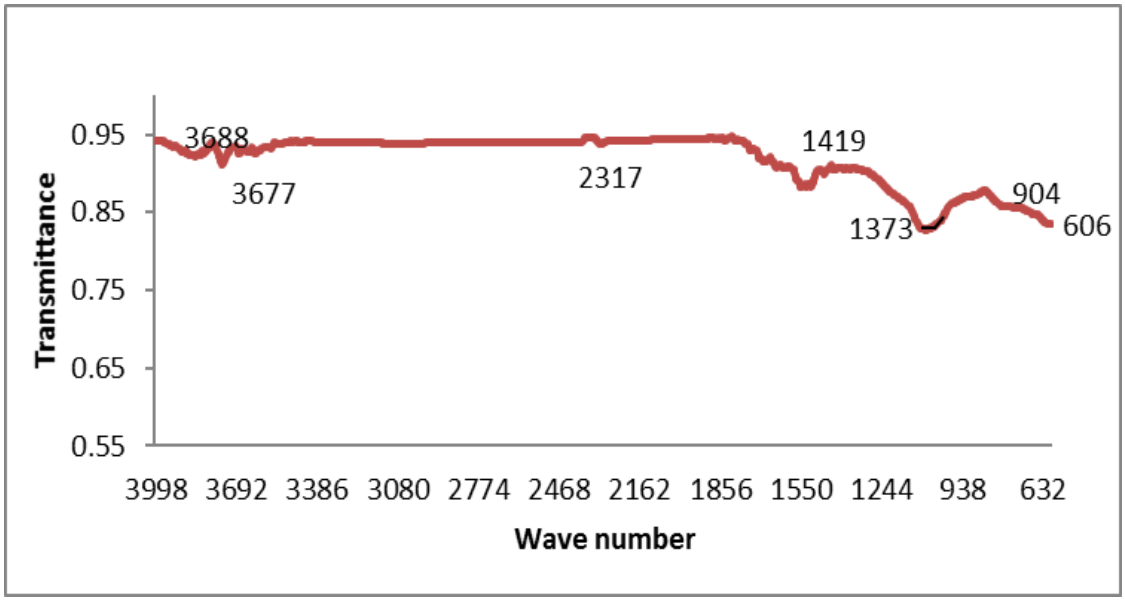


Figure 4.15 (a): FTIR spectra of 2.5 N H_3PO_4 Rice Bran Filtered

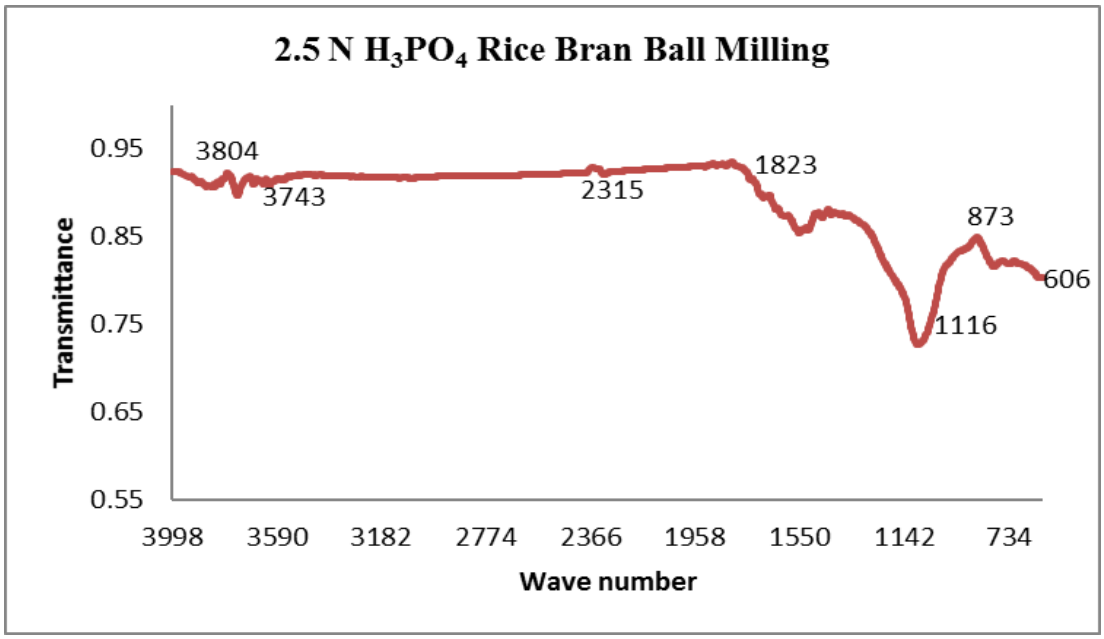


Figure 4.15 (b): FTIR spectra of 2.5 N H_3PO_4 Rice Bran ball milling

4.8.6 FTIR of 2.5 N H_2SO_4 Sesame activated carbon

The figure 4.16 (a and b) showed the FTIR spectra in 2.5 N H_2SO_4 sesame (filtered) at wavelength of 1381cm^{-1} assigned to stretching of C-OH i.e. phenolic and ethers (Kristianto *et al.*, 2017). In 2.5 N H_2SO_4 sesame (ball milling) **t**he FTIR spectra at peak 1619 cm^{-1} and 1134 cm^{-1} were assigned phenyl and C–H functional group and 611-714 cm^{-1} assigned C–O–C group (Mirghania et al., 2003).

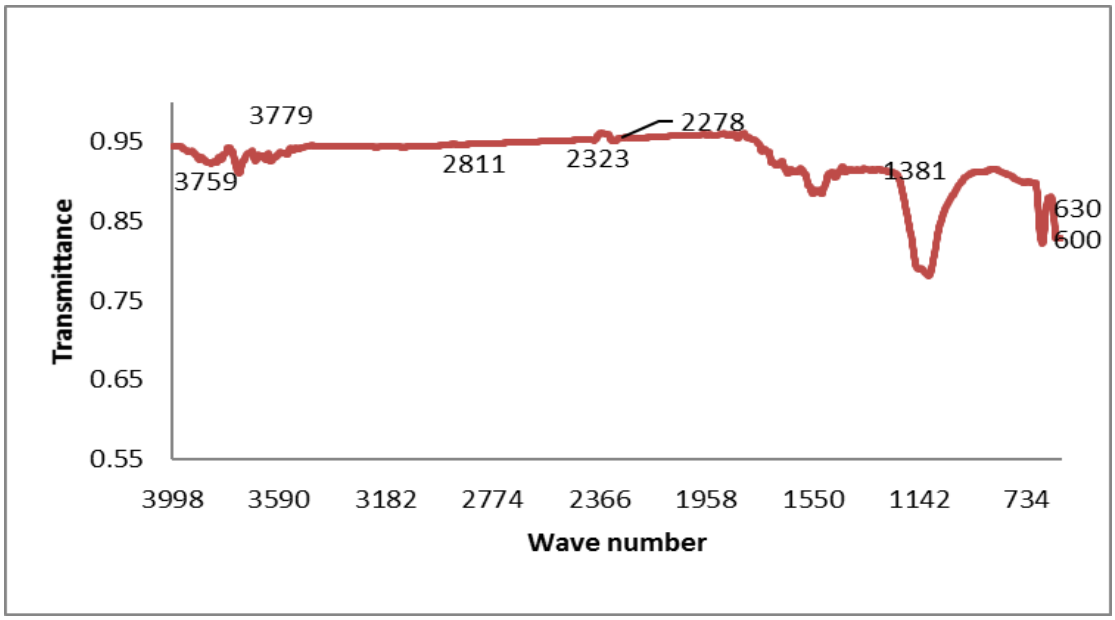


Figure 4.16 (a): FTIR spectra of 2.5 N H_2SO_4 Sesame Filtered

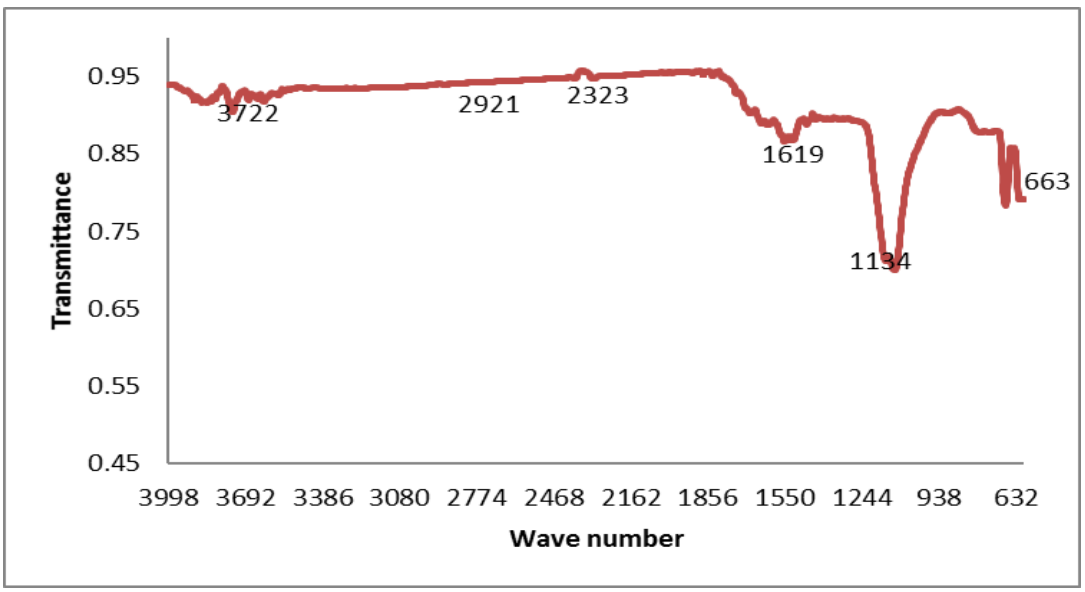


Figure 4.16 (b): FTIR spectra of 2.5 N H_2SO_4 Sesame ball milling

4.8.7 FTIR of Commercial activated carbon

The figure 4.17 showed the peaks at range from 3900 to 3743 cm^{-1} are due to the stretching vibration of O-H. The peak at 2345 cm^{-1} represented the symmetric C-C stretching of the functional group present on the activated carbon surface. The peak present at 1581 cm^{-1} represented the N-H (bending) and NO (stretch) stretching vibration of amide and nitro functional groups on the activated carbon surface (Hassan and Ahmed, 2011).

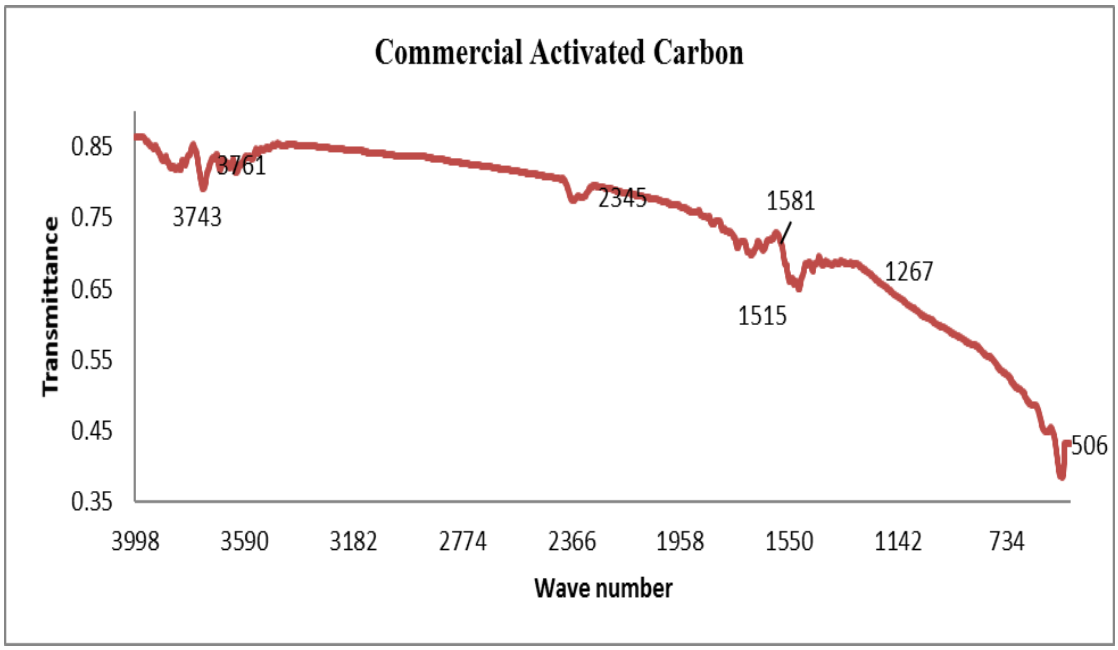


Figure 4.17: FTIR spectra of commercial activated carbon

4.9 Absorbency of Acid dye effluent

4.9.1 Absorbency of Acid dye effluent using 2.5 N H_3PO_4 Almond activated carbon

Table 4.10: Absorbency of 2.5 N H_3PO_4 Almond activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.5%	1.140	1.150	1.133	1.130	1.150	1.120	0.153	0.120	0.113	**0.801**	30	**0.570**
1.0%	1.077	0.987	1.033	0.870	0.860	0.840	0.043	0.403	0.040	**0.644**	60	**0.526**
1.5%	0.993	0.857	0.783	0.503	0.430	0.360	0.033	0.043	0.050	**0.450**	90	**0.508**
2.0%	0.673	0.0527	0.477	0.183	0.107	0.107	0.037	0.037	0.040	**0.243**		
Mean of Activated carbon	**0.903**			**0.638**			**0.063**					

	S. Ed(±)	**CD (0.05)**
Activated carbon	**0.0040**	**0.0081**
Time	**0.0040**	**0.0081**
Concentration	**0.0047**	**0.0093**
Activated carbon*time	**0.0070**	**0.0140**
Activated carbon* concentration	**0.0081**	**0.0161**
Time*concentration	**0.0081**	**0.0161**
Activated carbon* time*concentration	**0.0140**	**0.0280**

Foot note:- Data given in the table are mean of three replication

Table 4.10 and Figure 4.18 (a-d) indicated that the adsorption capacity was decreased with the increased of activated carbon. It was noticed that at a low amount of activated carbon, high absorbency uptake was obtained. This result indicated that more surface area was made due to the increased mass of activated carbon. In addition to this, the table also revealed that among all the concentrations, absorbency was decreased at 2.0% activated carbon. The low absorbency was recorded in case of 2.5 N H_3PO_4 almond (filtered) activated carbon, treated in different time periods at different concentrations. From the statistical analysis, it was clear that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 almond activated carbon.

The lowest absorbency (1.133) was recorded in 90 minute at 0.5% concentration while the absorbency (0.987) was recorded in 60 minute at 1.0% concentration followed by (0.783) in 90 minutes at 1.5% concentration, and the absorbency (0.477) in 90 minute at 2.0% concentration respectively. In 2.5 N H_3PO_4 almond (ball milling) activated carbon, the lowest absorbency (1.120) was recorded in 0.5% concentration at 90 minute time period while the absorbency (0.840) was recorded in 1.0% concentration at 90 minute time period followed by (0.360) in 1.5% concentration at 90 minute time period and (0.107) in 2.0% concentration at 60 minute and 90 minute time period respectively. In case of commercial activated carbon, the lowest absorbency was recorded (0.113) in 90 minute at 0.5% concentration followed by (0.040) in 90 minute at 1.0% concentration, (0.33) in 30 minute at 1.5% concentration and (0.037) in 30 minute and 60 minute at 2.0% concentration. The lowest value for the mean of concentration (2.0%) and the mean of activated carbon was recorded as 0.243 and 0.063 respectively in commercial activated carbon. The lowest value for the mean of time was recorded as 0.508 against 90 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in ball milling (0.638).

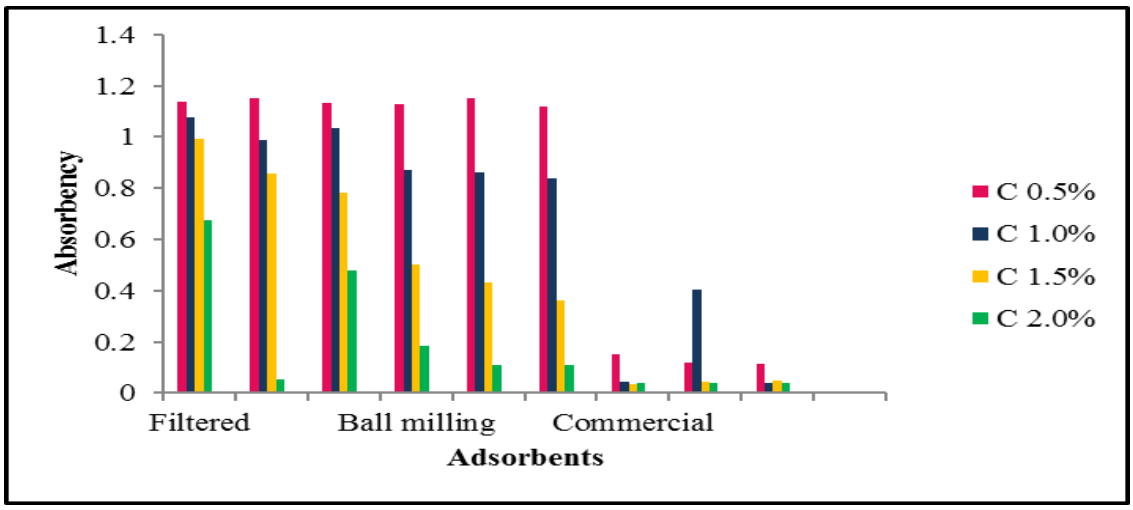


Figure 4.18 (a): Absorbency of (2.5 N H_3PO_4 Almond) activated carbon

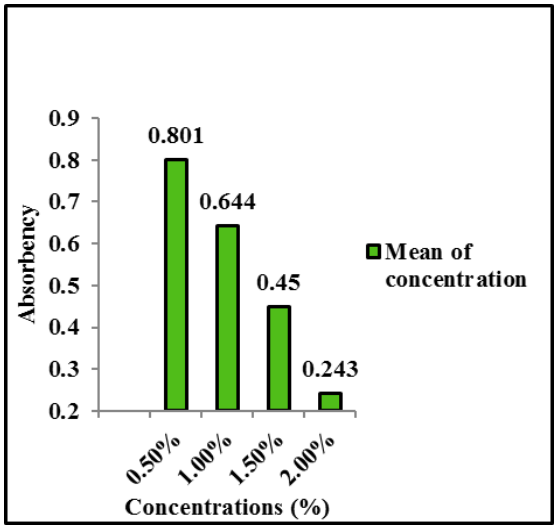


Figure 4.18 (b): Mean of concentrations

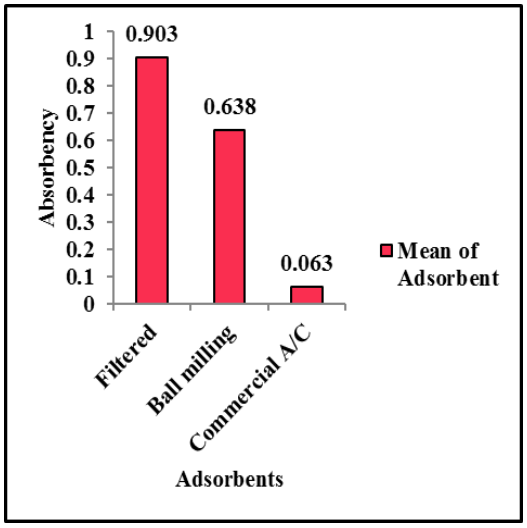


Figure 4.18 (c): Mean of activated carbons

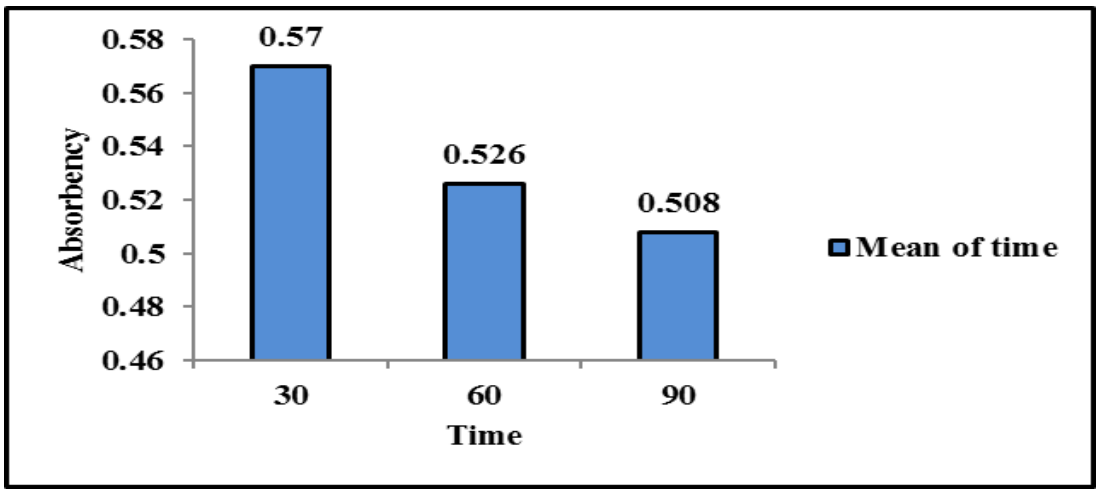


Figure 4.18 (d): Mean of time

(Akpen *et al.*, 2017) have studied on activated carbon from a local variety of mango (*Magnifera indica)* by chemical activation with Zinc Chloride ($ZnCl_2$) before and after carbonization found that the mango seed endocarp activated carbon (MSEAC) can be used effectively to remove color from textile wastewater at the optimum conditions and it proves the best impregnation ratio produced as 1:4 and the color removal was 86.3% in 40 min. It might be due to increase in the quantity of activated carbon results in a corresponding increase in the amount of color removed, which is as a result of increase in surface area and hence more sites were available for adsorption of colour. Similar results were obtained by Namasivayam and Yamuna, 2007 Akpen *et al.*, 2011.

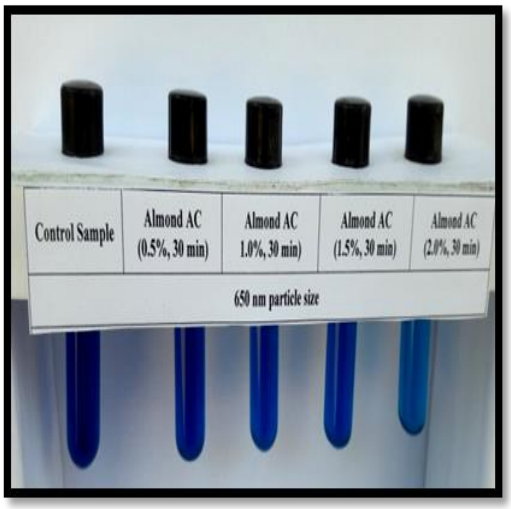


Plate 4.1 (a): 30 minute

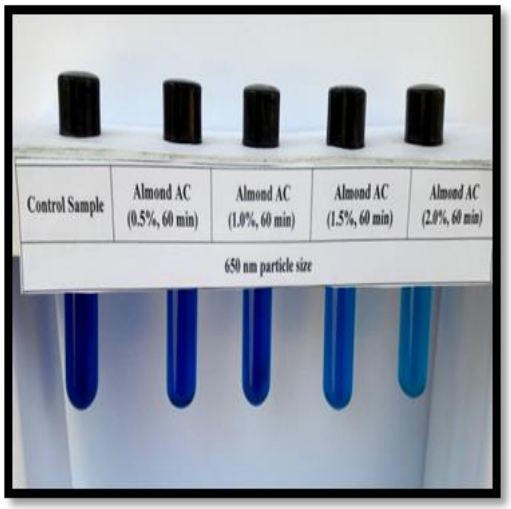


Plate 4.1 (b): 60 minute

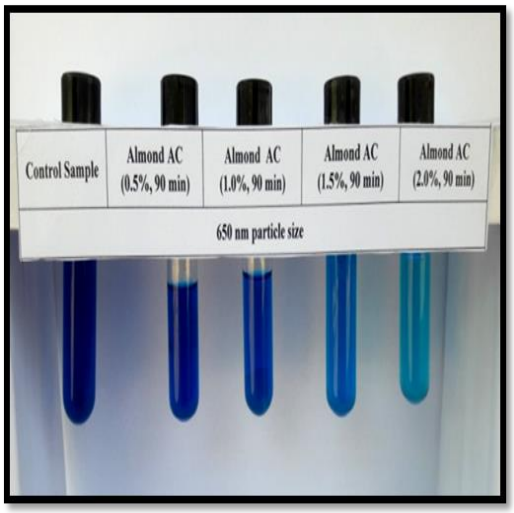


Plate 4.1 (c): 90 minute

Plate 4.1: 2.5 N H_3PO_4 Almond (Filtered) activated carbons at different time

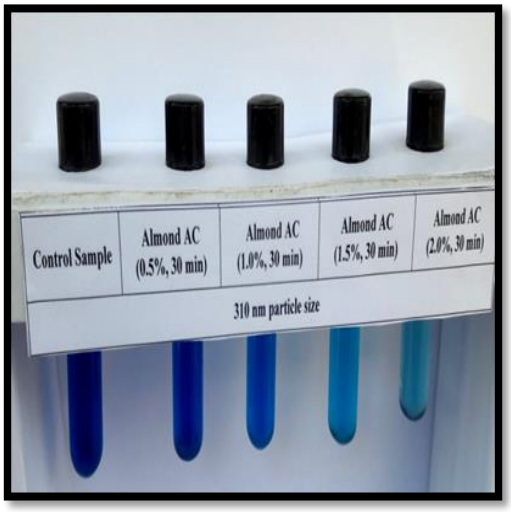


Plate 4.2 (a): 30 minute

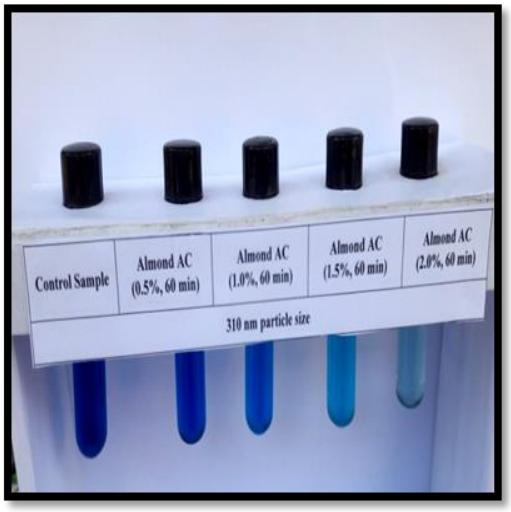


Plate 4.2 (b): 60 minute

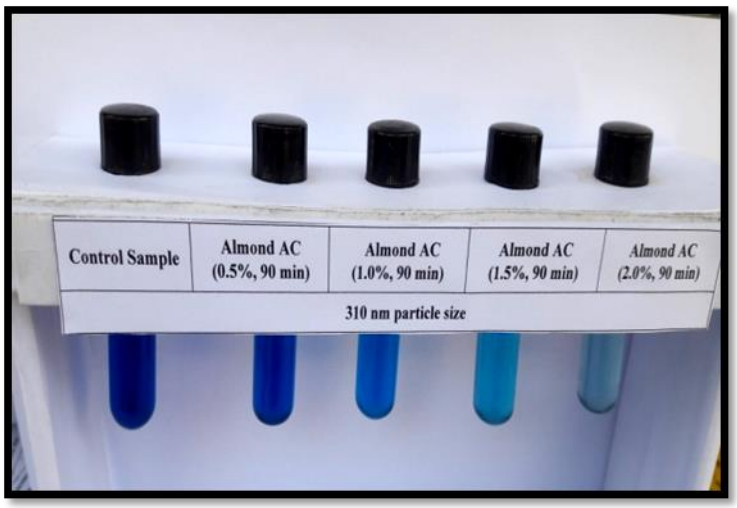


Plate 4.2 (c): 90 minute

PLATE 4.2: 2.5 N H_3PO_4 Almond (ball milling) activated carbons at different time

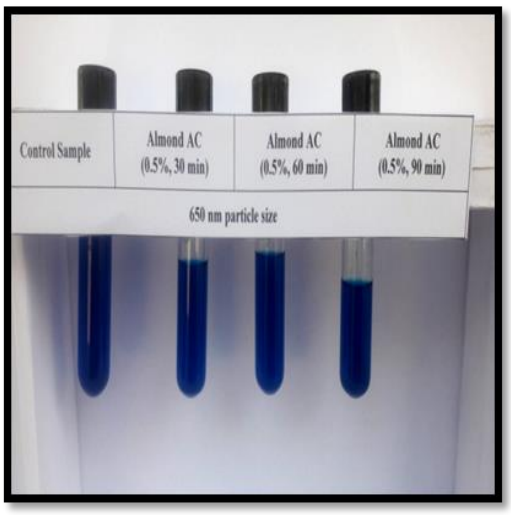


Plate 4.3 (a): 0.5% Concentration

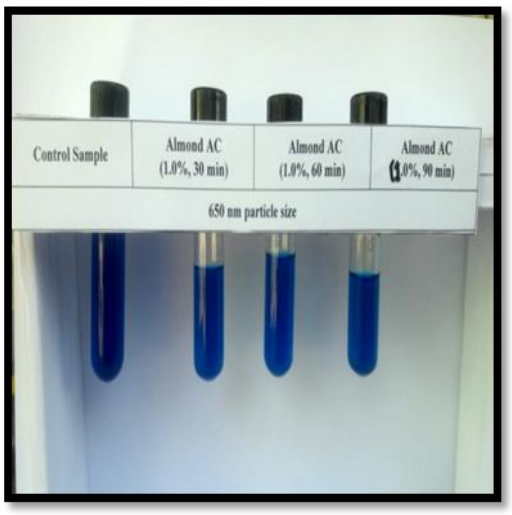


Plate 4.3 (b): 1.0% Concentration

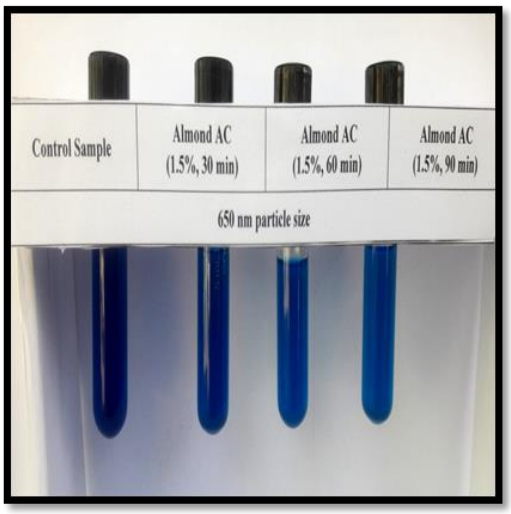


Plate 4.3 (c): 1.5% Concentration

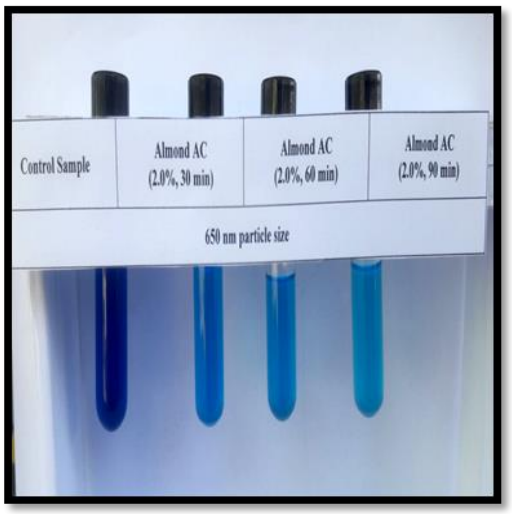


Plate 4.3 (d): 2.0% Concentration

PLATE 4.3: 2.5 N H_3PO_4 Almond (Filtered) activated carbons at different concentrations

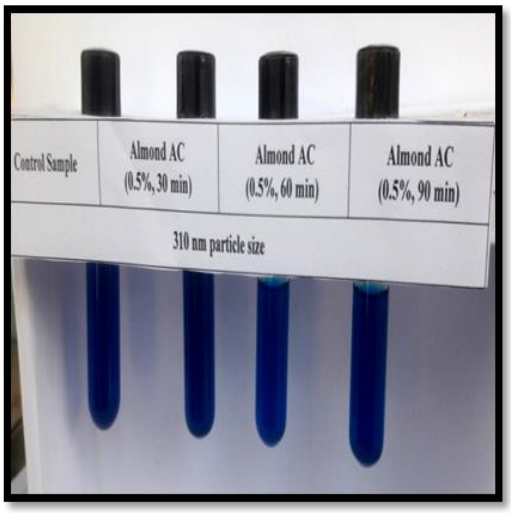


Plate 4.4 (a): 0.5% Concentration

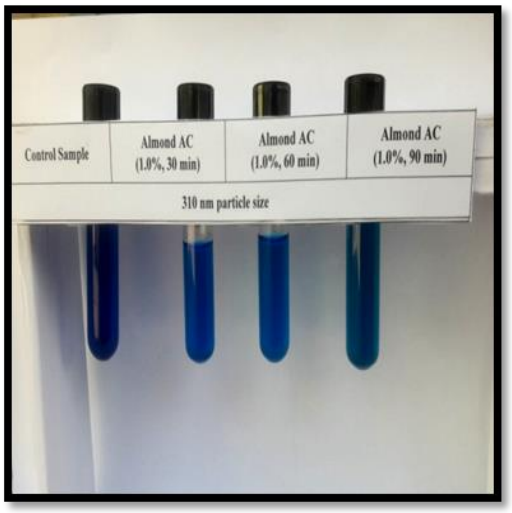


Plate 4.4 (b): 1.0% Concentration

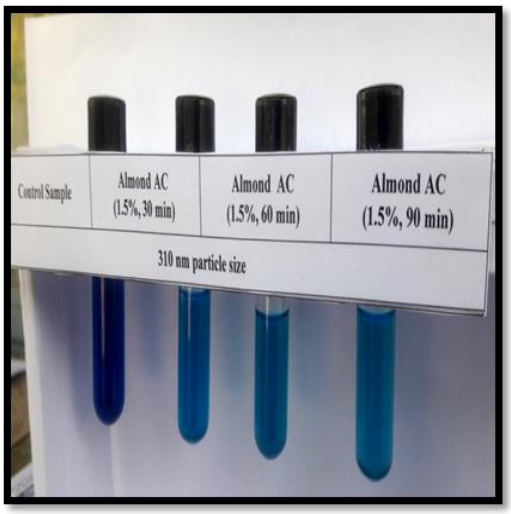


Plate 4.4 (c): 1.5% Concentration

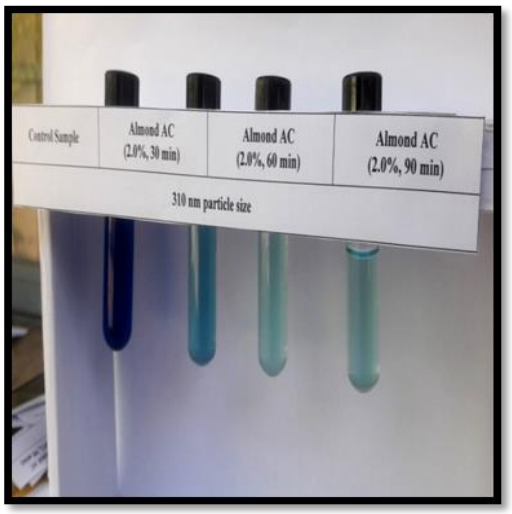


Plate 4.4 (d): 2.0% Concentration

PLATE 4.4: 2.5 N H_3PO_4 Almond (ball milling) activated carbons at different concentrations

4.9.2 Removal of Acid dye effluent using 1 N H_3PO_4 Coconut activated carbon

Table 4.11: Absorbency of 1 N $_3PO_4$ Coconut activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.5%	1.147	1.087	1.067	1.277	1.223	1.290	0.153	0.120	0.113	**0.831**	30	**0.562**
1.0%	1.013	0.940	0.847	0.967	0.953	0.943	0.043	0.043	0.040	**0.643**	60	**0.504**
1.5%	0.653	0.620	0.350	0.713	0.527	0.500	0.33	0.043	0.050	**0.388**	90	**0.465**
2.0%	0.383	0.227	0.140	0.323	0.227	0.197	0.037	0.037	0.040	**0.179**		
Mean of Activated carbon	**0.706**			**0.762**			**0.063**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0058**	**0.0116**
Time	**0.0058**	**0.0116**
Concentration	**0.0067**	**0.0135**
Activated carbon*time	**0.0101**	**0.0202**
Activated carbon* concentration	**0.0117**	**0.0233**
Time*concentration	**0.0117**	**0.0233**
Activated carbon* time*concentration	**0.0202**	**0.0404**

Foot note:- Data given in the table are mean of three replication

Table 4.11 and Figure 4.19 (a-d) depicted that the adsorption capacity was decreased with the increment of the activated carbon. It was noticed that at a low amount of activated carbon, high absorbency uptake was obtained. This result indicated that more surface area was made due to the increased mass of activated carbon. In addition to this, the table also revealed that among all the concentrations, absorbency was decreased at 2.0% activated carbon. From the statistical analysis, it was clear that all the factors and their interactions were found to be significant at 5% level with respect to 1 N H_3PO_4 coconut activated carbon.

The low absorbency was recorded in case of 1 N H_3PO_4 coconut (filtered) activated carbon treated in different time periods at different concentrations. The lowest absorbency (1.067) were recorded in 90 minute at 0.5% concentration followed by (0.847) in 90 minute at 1.0% concentration while the absorbency (0.350) in 90 minute at 1.5% concentration and (0.140) in 90 minute at 2.0% concentration respectively. In 1 N H_3PO_4 coconut (ball milling) activated carbon, the lowest absorbency (1.223) at 0.5% concentration in 60 minute followed by (0.943) at 1.0% concentration in 90 minute, (0.500) at 1.5% concentration in 90 minute and (0.197) at 2.0% concentration in 90 minute of time period respectively. In case of commercial activated carbon the lowest absorbency were recorded (0.113) in 90 minute at 0.5% concentration followed by (0.040) in 90 minute at 1.0% concentration, (0.33) in 30 minute at 1.5% concentration and (0.037) in 30 minute and 60 minute of time period at 2.0% concentration respectively. The lowest value for the mean of concentration (2.0%) and the mean of activated carbon was recorded as 0.179 and 0.063 respectively in commercial activated carbon. The lowest value for the mean of time was recorded as 0.465 against 90 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in filtered (0.706).

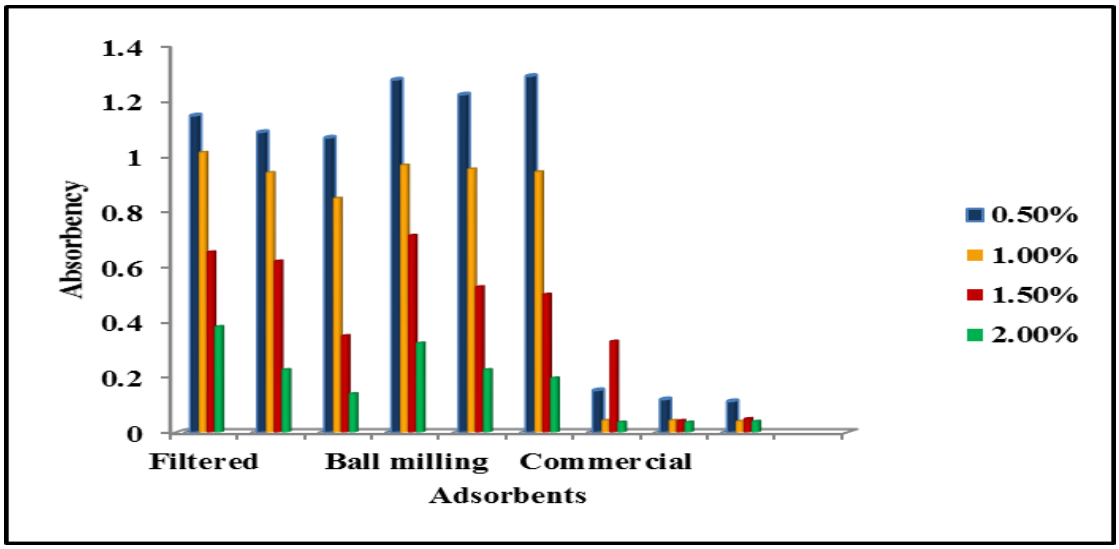


Figure 4.19 (a): Absorbency of (1 N H_3PO_4 Coconut) activated carbon

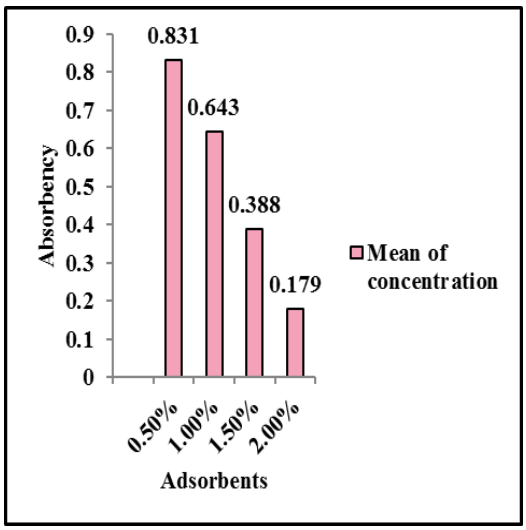


Figure 4.19 (b): Mean of concentration

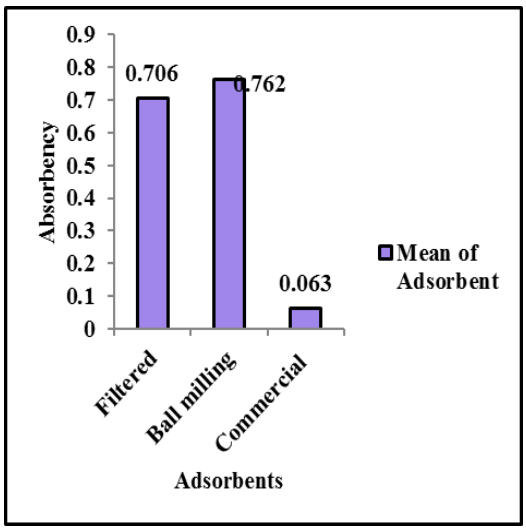


Figure 4.19 (c): Mean of activated carbon

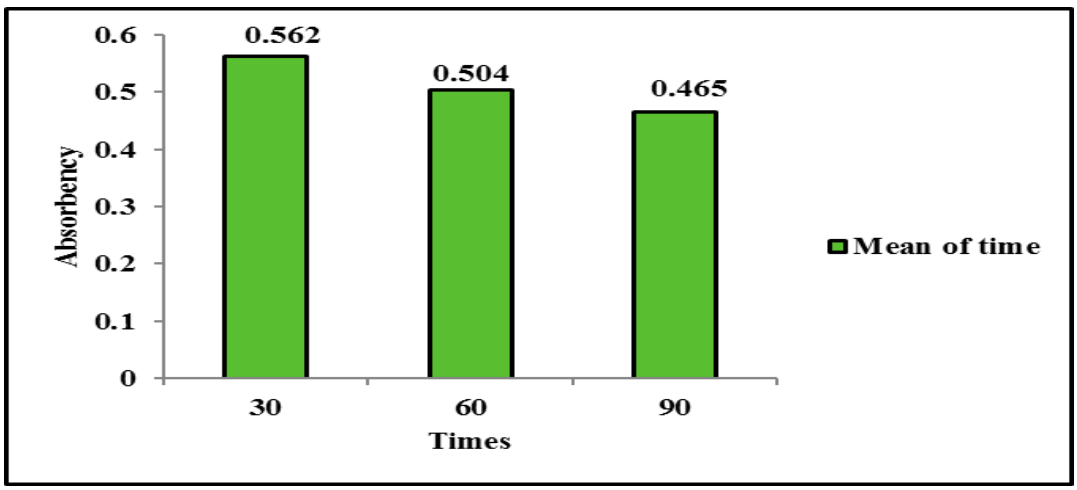


Figure 4.19 (d): Mean of time

Baskaralingam *et al.,* (2006) studied the effectiveness of the prepared organobentonite viz. cetyldimethylbenzylammonium chloride (CDBA) and cetylpyridinium chloride (CP) in removing of Acid Red 151 (AR 151) from aqueous dye solution found that the CDBA-bentonite and CP-bentonite are good activated carbons for the removal of colour with adsorption capacity 357.14 mg g^{-1} and 416.66 mg g^{-1} respectively. This is due to the mass of the activated carbon dosages that increased with the increase in percentage of dye removal. Baskaralingam *et al.,* (2006) reported that with high dosage of the activated carbon there was an increase in surface area.

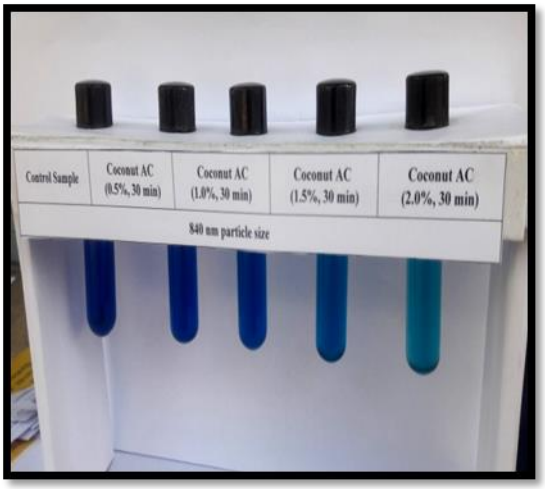


Plate 4.5 (a): 30 minute

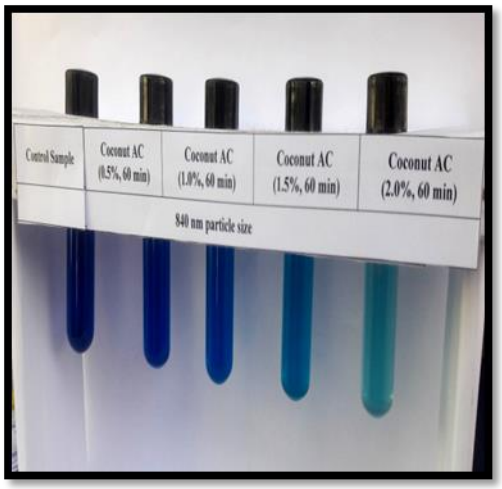


Plate 4.5 (b): 60 minute

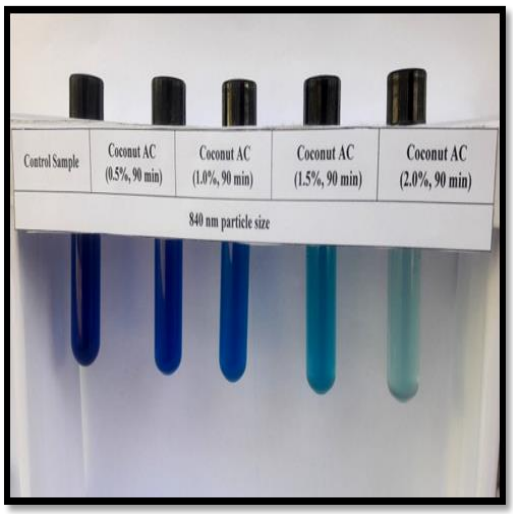


Plate 4.5 (c): 90 minute

PLATE 4.5: 1 NH_3PO_4Coconut (Filtered) activated carbons at different time

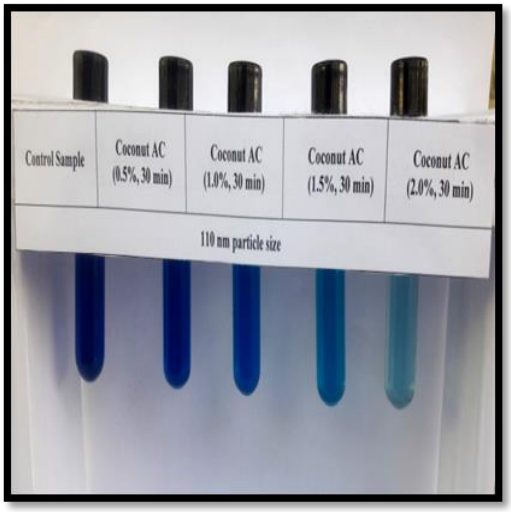


Plate 4.6 (a): 30 minute

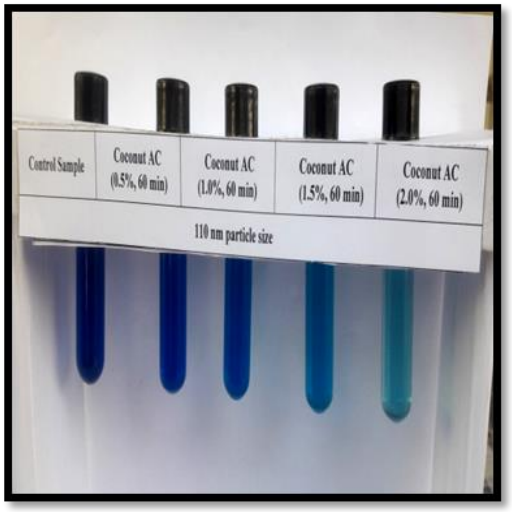


Plate 4.6 (b): 60 minute

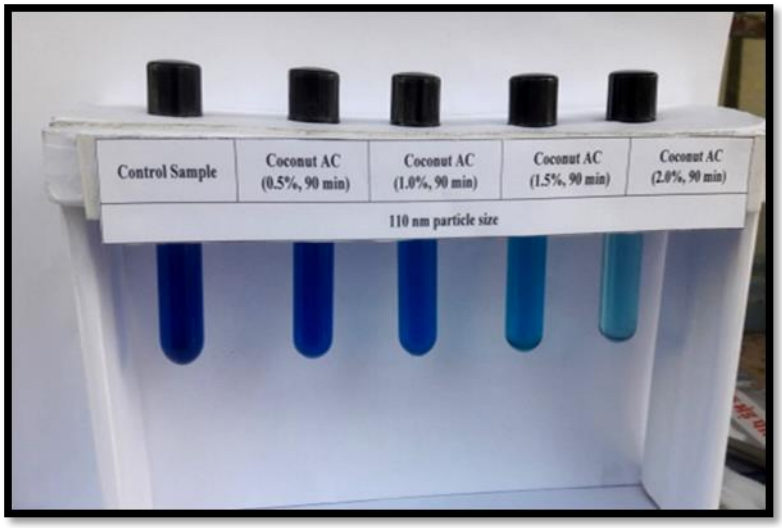


Plate 4.6 (c): 90 minute

PLATE 4.6: 1 NH_3PO_4Coconut (ball milling) activated carbons at different time

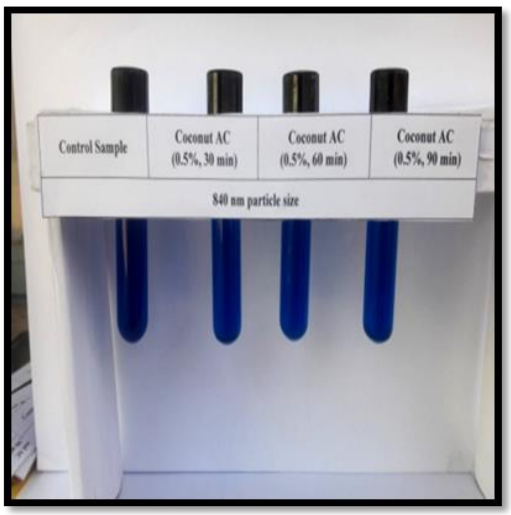


Plate 4.7 (a): 0.5% Concentration

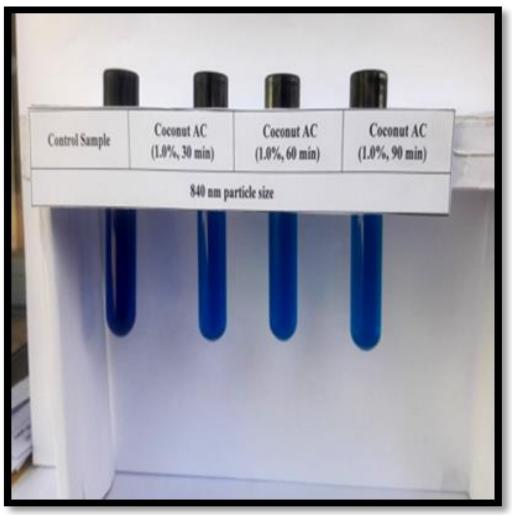


Plate 4.7 (b): 1.0% Concentration

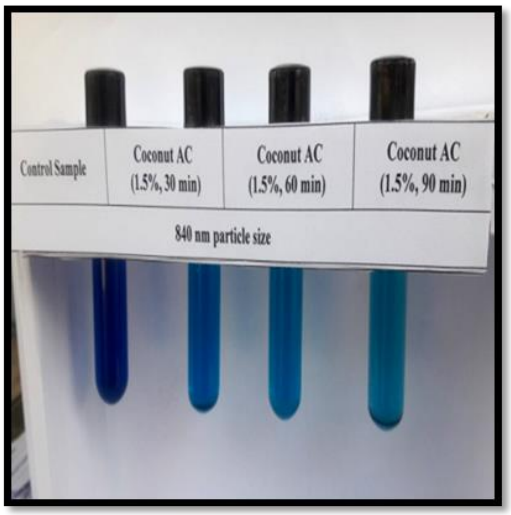


Plate 4.7 (c): 1.5% Concentration

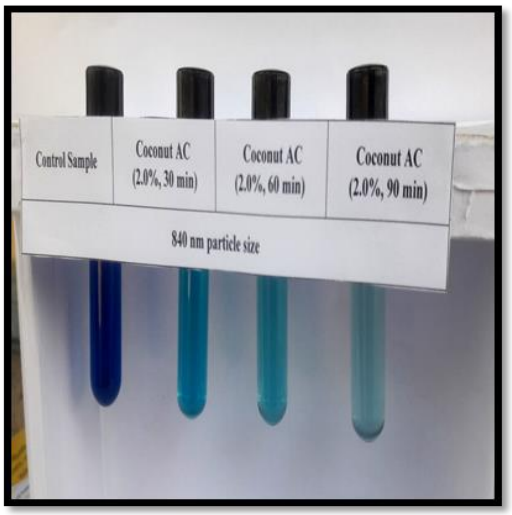


Plate 4.7 (d): 2.0% Concentration

PLATE 4.7: 1 NH_3PO_4Coconut (Filtered) activated carbons at different concentration

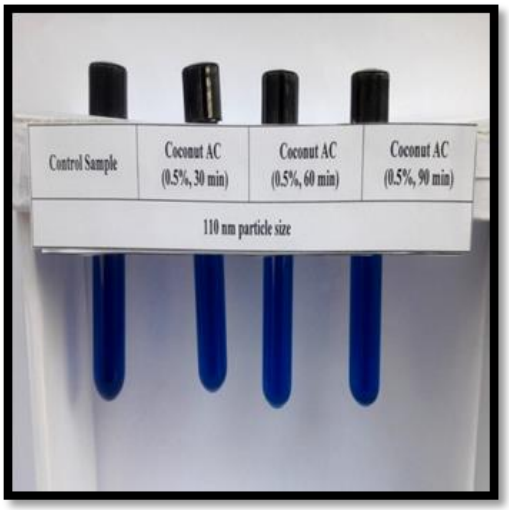


Plate 4.8 (a): 0.5% Concentration

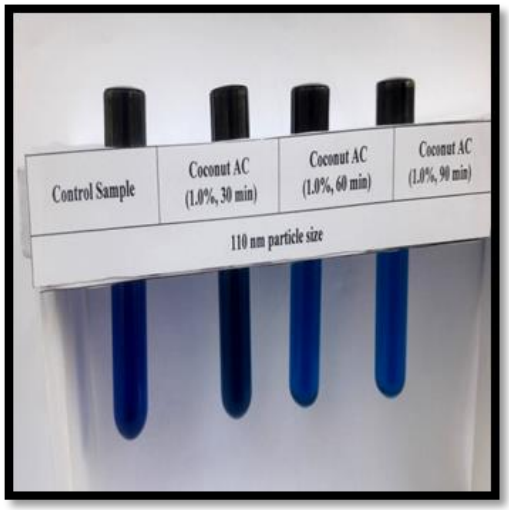


Plate 4.8 (b): 1.0% Concentration

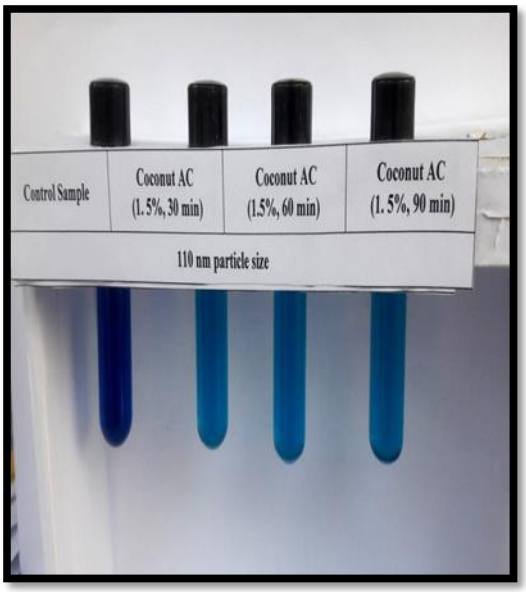


Plate 4.8 (c): 1.5% Concentration

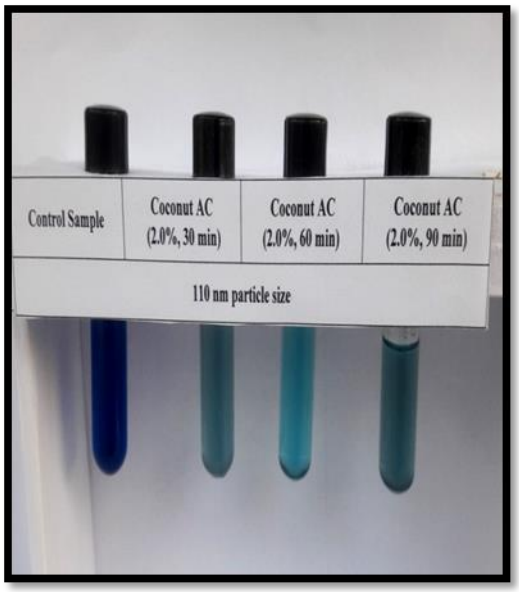


Plate 4.8 (d): 2.0% Concentration

PLATE 4.8: 1 NH_3PO_4Coconut (ball milling) activated carbons at different concentrations

4.9.3 Removal of Acid dye effluent using 2.5 N H_3PO_4 Mustard activated carbon

Table 4.12: Absorbency of 2.5 N H_3PO_4 Mustard activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.5%	1.283	1.260	1.230	1.117	1.193	1.113	0.153	0.120	0.113	**0.843**	30	**0.645**
1.0%	1.217	1.190	1.167	0.867	0.860	0.850	0.043	0.043	0.040	**0.697**	60	**0.627**
1.5%	1.123	1.070	1.057	0.547	0.493	0.477	0.33	0.043	0.050	**0.544**	90	**0.601**
2.0%	1.053	0.997	0.937	0.263	0.213	0.133	0.037	0.037	0.040	**0.412**		
Mean of Activated carbon	**1.132**			**0.677**			**0.063**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0050**	**0.0100**
Time	**0.0050**	**0.0100**
Concentration	**0.0058**	**0.0116**
Activated carbon*time	**0.0087**	**0.0174**
Activated carbon* concentration	**0.0101**	**0.0201**
Time*concentration	**0.0101**	**0.0201**
Activated carbon* time*concentration	**0.0175**	**0.0348**

Foot note:- Data given in the table are mean of three replication

Similar trends were observed with decreasing adsorption capacity in table 4.12 and figure 4.20 (a-d) and absorbency was decreased at 2.0% activated carbon. From the statistical analysis, it indicated that all the factors and their interactions were found to be significantly influence at 5% level with respect to 2.5 N H_3PO_4 mustard activated carbon.

The low absorbency was recorded in case of 2.5 N H_3PO_4 mustard (filtered) activated carbon treated in different time periods at different concentrations. The lowest absorbency were recorded (1.230), (1.167), (1.057) and (0.937) respectively in 90 minute of contact time at 0.5%, 1.0%, 1.5% and 2.0% concentration respectively. In 2.5 N H_3PO_4 mustard (ball milling) activated carbon the lowest absorbency were recorded (1.113) at 0.5% concentration in 90 minute followed by (0.850) at 1.0% concentration in 90 minute, while the absorbency (0.477) at 1.5% concentration in 90 minute and (0.133) at 2.0% concentration in 90 minute of time period. In commercial activated carbon and, the lowest absorbency were recorded (0.113) at 0.5% concentration and a (0.040) at 1.0% concentration in 90 minute of contact time followed by (0.33) at 1.5% concentration in 30 minute and (0.037) at 2.0% concentration in 30 minute and 60 minute of contact time respectively. The lowest value for the mean of concentration (2.0%) and the mean of activated carbon was recorded as 0.412 and 0.063 respectively in commercial activated carbon. The lowest value for the mean of time was recorded as 0.601 against 90 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in ball milling (0.677).

(Ahmadi *et al.,* 2020) studied on the model with the aim of reduction of Acid Blue 92 (AB 92) dye from aqueous solutions by adsorption onto synthesized neodymium (III) oxide (Nd_2O_3) nanoparticles. They have opined that the adsorption efficiency declined with increasing initial concentration which might be due to the higher concentrations of the removal efficiency. Roy, et al., 2012 have studied on adsorption of anionic-azo dye from aqueous solution by lignocellulose-biomass jute fiber and found that due to the high concentrations saturation of the activated carbon surface with the adsorbate contaminant decreases.

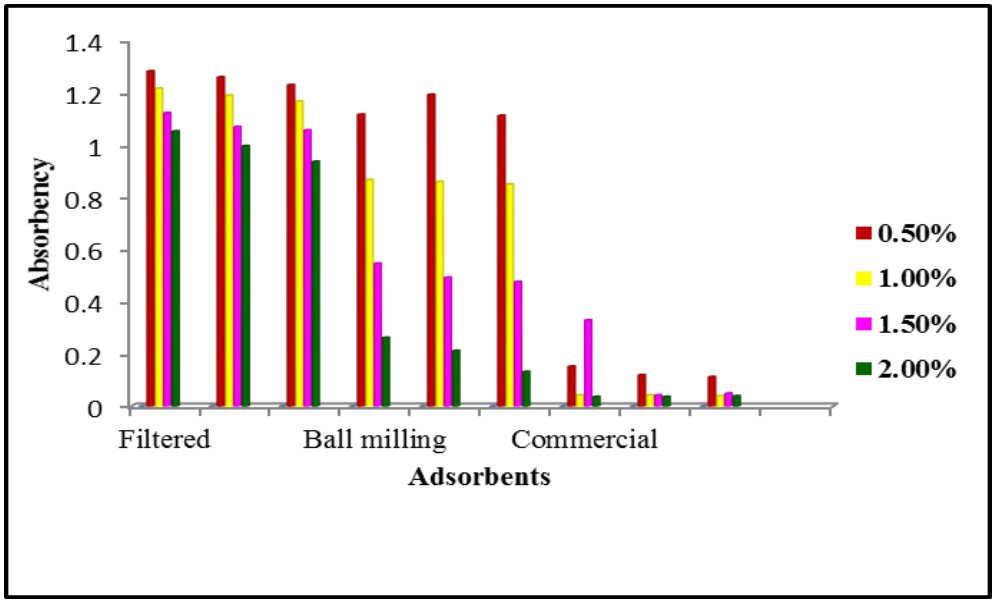


Figure 4.20 (a): Absorbency of (2.5 N H_3PO_4 Mustard) activated carbon

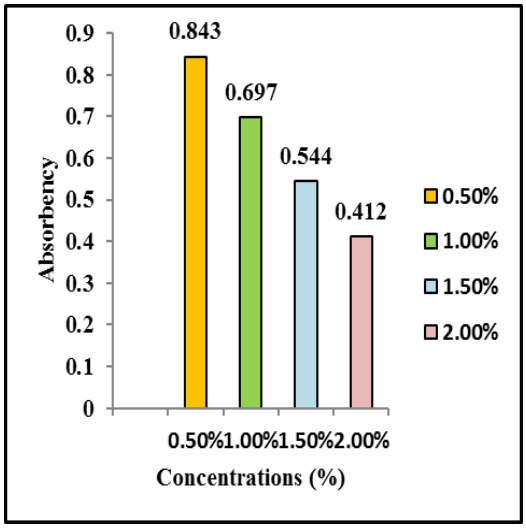


Figure 4.20 (b): Mean of concentration

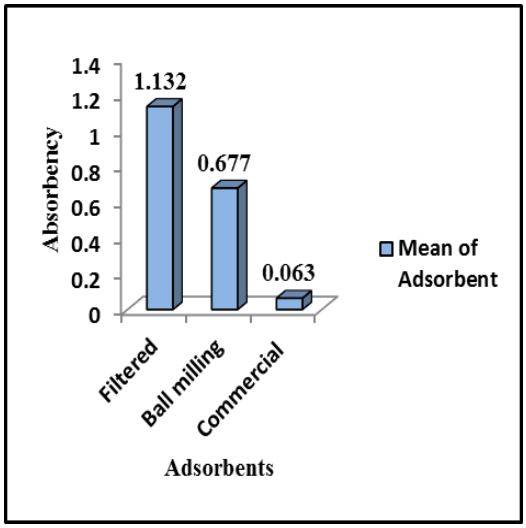


Figure 4.20 (c): Mean of activated carbon

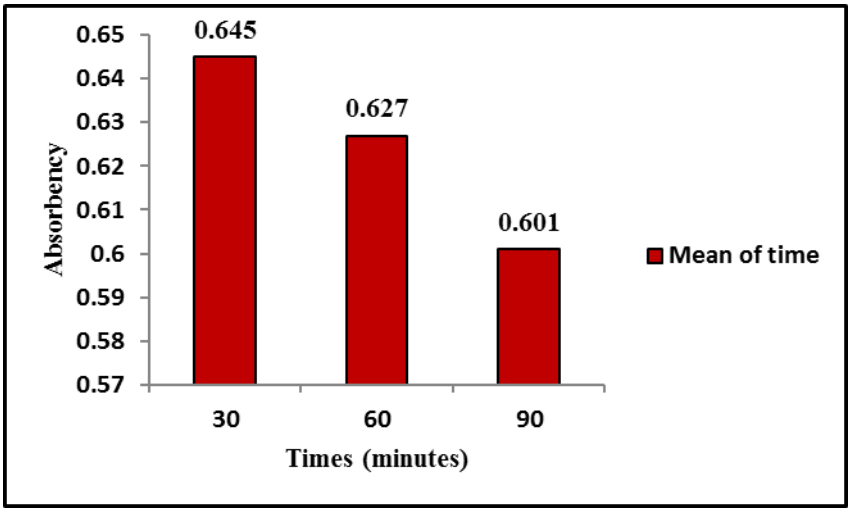


Figure 4.20 (d): Mean of time

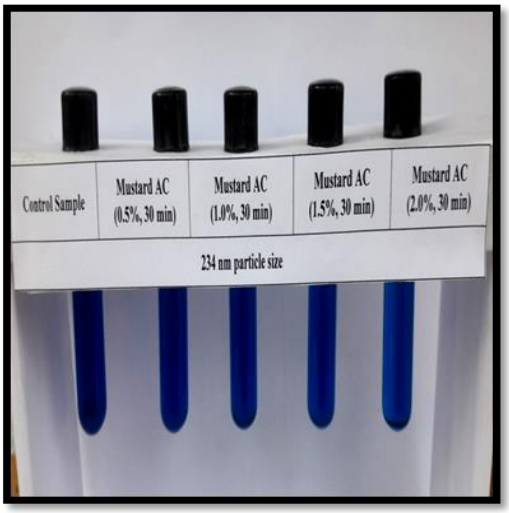


Plate 4.9 (a): 30 minute

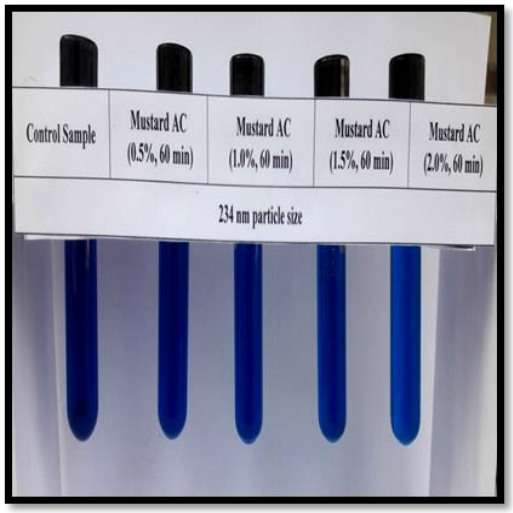


Plate 4.9 (b): 60 minute

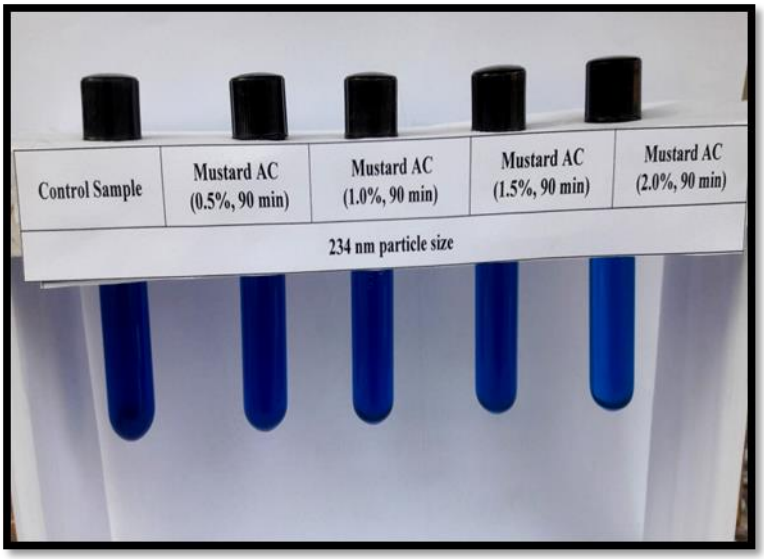


Plate 4.9 (c): 90 minute

PLATE 4.9: 2.5 N H_3PO_4 Mustard (Filtered) activated carbons at different time

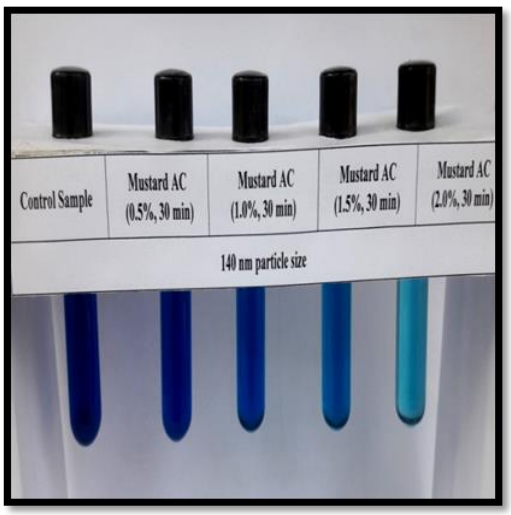


Plate 4.10 (a): 30 minute

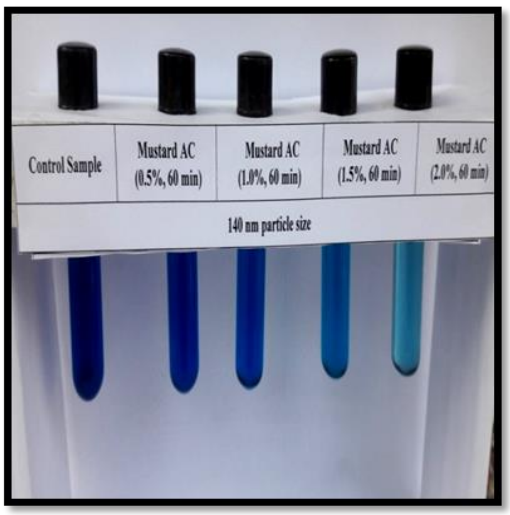


Plate 4.10 (b): 60 minute

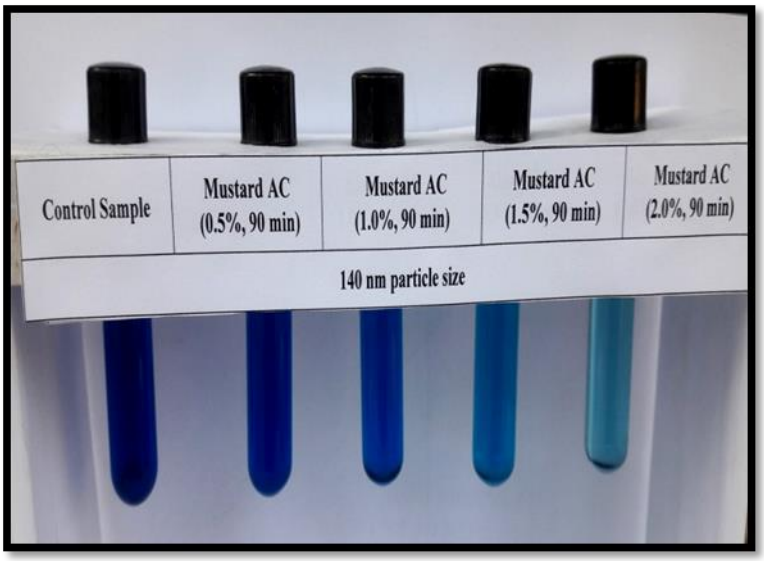


Plate 4.10 (c): 90 minute

PLATE 4.10: 2.5 N H_3PO_4 Mustard (ball milling) activated carbons at different time

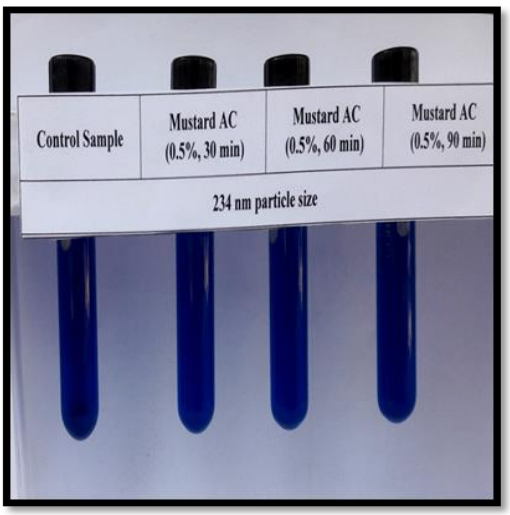


Plate 4.11 (a): 0.5% Concentration

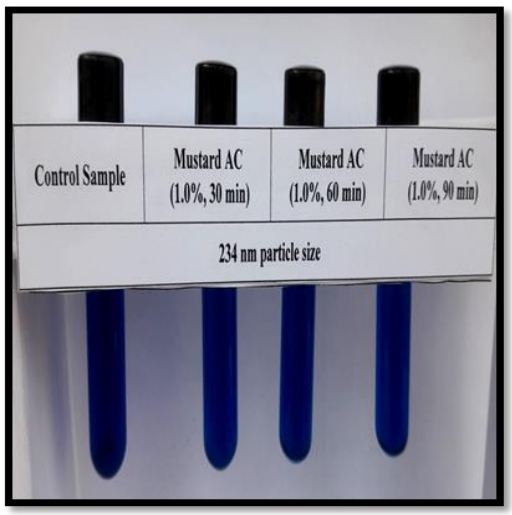


Plate 4.11 (b): 1.0% Concentration

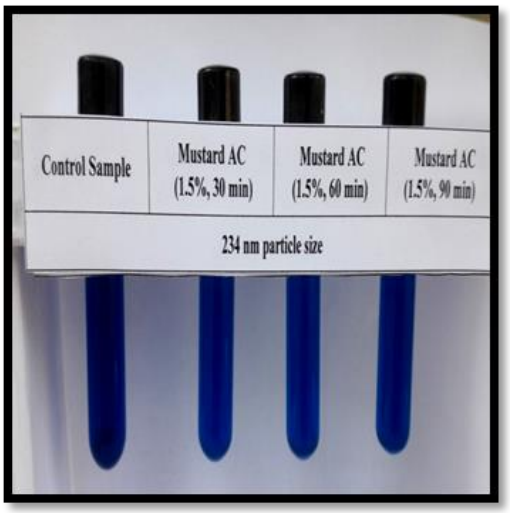


Plate 4.11 (c): 1.5% Concentration

Plate 4.11 (d): 2.0% Concentration

PLATE 4.11: 2.5 N H_3PO_4 Mustard (Filtered) activated carbons at different concentrations

Plate 4.12 (a): 0.5% Concentration

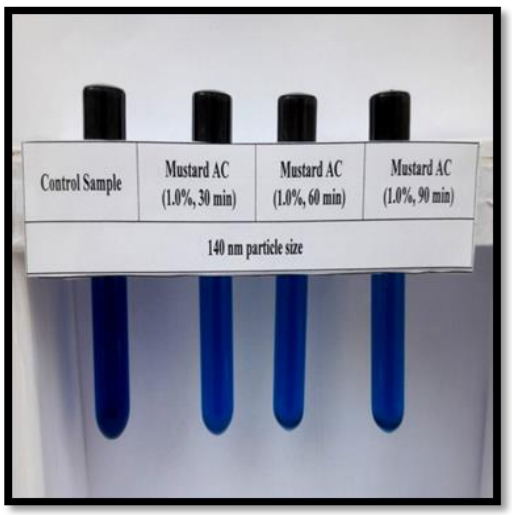


Plate 4.12 (b): 1.0% Concentration

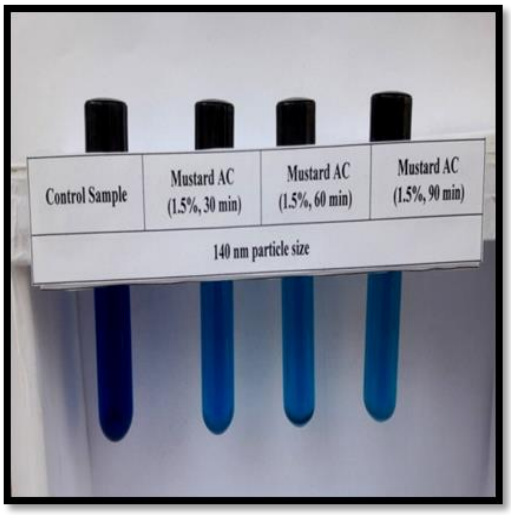


Plate 4.12 (c): 1.5% Concentration

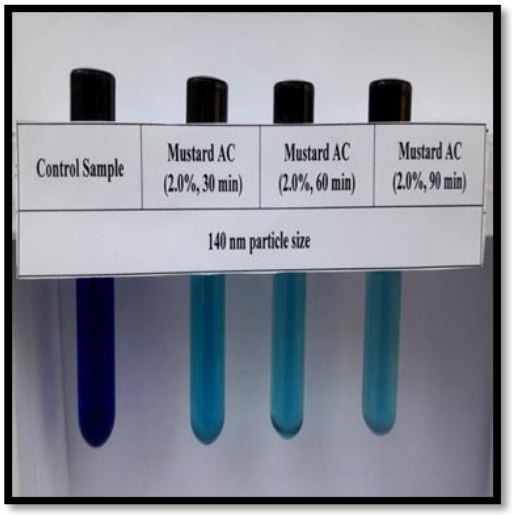


Plate 4.12 (d): 2.0% Concentration

PLATE 4.12: 2.5 N H_3PO_4 Mustard (ball milling) activated carbons at different concentrations

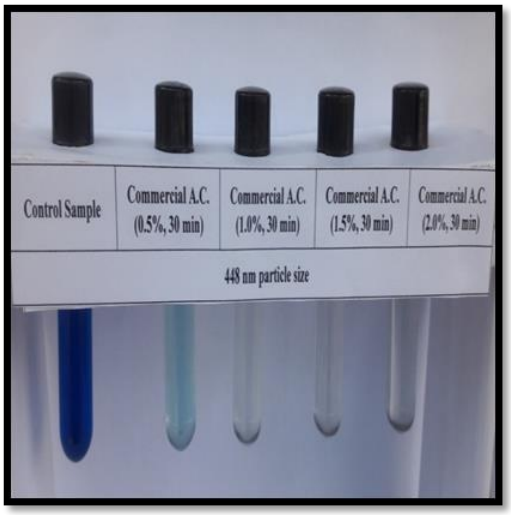


Plate 4.13 (a): 30 minute

Plate 4.13 (b): 60 minute

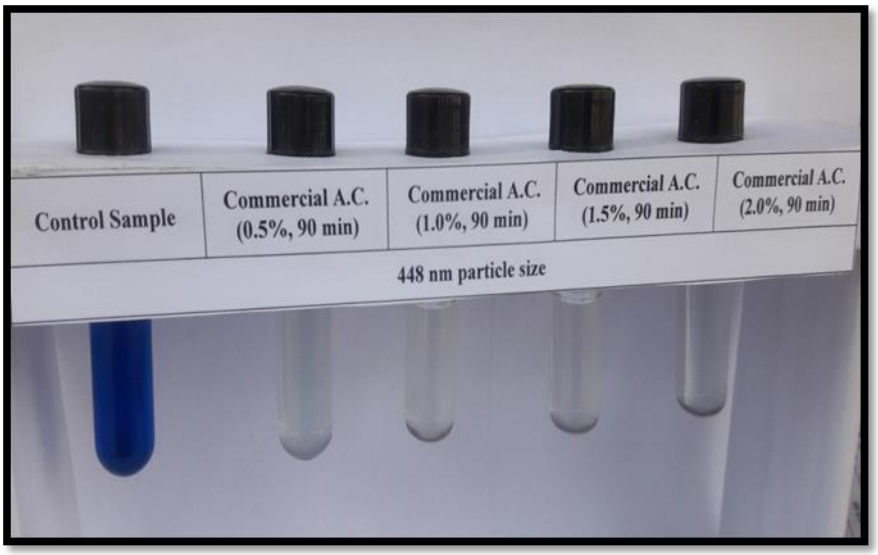


Plate 4.13 (c): 90 minute

Plate 4.13: Commercial activated carbons at different time

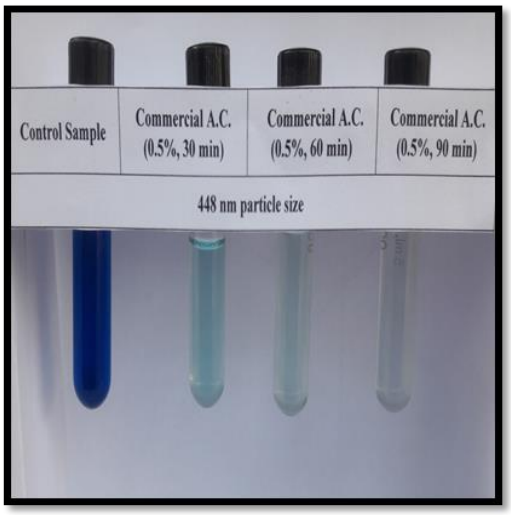


Plate 4.14 (a): 0.5% Concentration

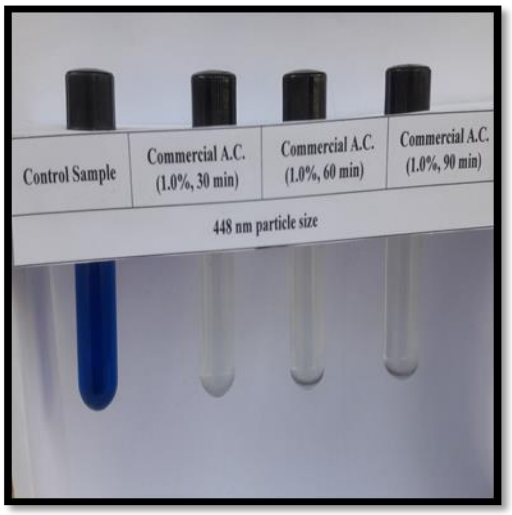


Plate 4.14 (b): 1.0% Concentration

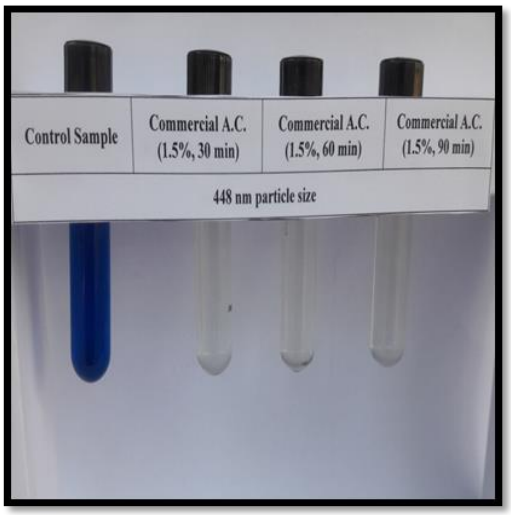


Plate 4.14 (c): 1.5% Concentration

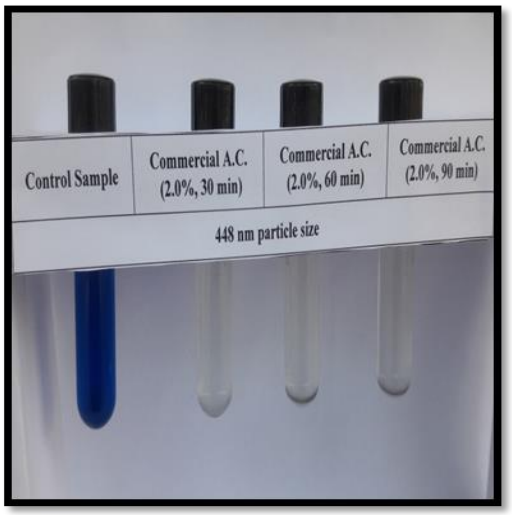


Plate 4.14 (d): 2.0% Concentration

PLATE 4.14: Commercial activated carbons at different concentrations

4.10 Absorbency of Metal complex dye effluent

4.10.1 Removal of Metal complex dye effluent using 2.5 N H_2SO_4 Almond activated carbon

Table 4.13: Absorbency of 2.5 N H_2SO_4 Almond activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.1%	0.683	0.680	0.690	0.863	0.833	0.670	0.387	0.390	0.450	**0.627**	30	**0.368**
0.25%	0.617	0.633	0.523	0.530	0.727	0.600	0.150	0.160	0.150	**0.454**	60	**0.393**
0.5%	0.500	0.500	0.507	0.267	0.397	0.353	0.017	0.013	0.007	**0.284**	90	**0.365**
1.0%	0.257	0.227	0.267	0.147	0.147	0.153	0.003	0.010	0.007	**0.135**		
Mean of Activated carbon	**0.507**			**0.474**			**0.145**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0123**	**0.0246**
Time	**0.0123**	**0.0246**
Concentration	**0.0142**	**0.0284**
Activated carbon*time	**0.0213**	**0.0425**
Activated carbon* concentration	**0.0246**	**0.0491**
Time*concentration	**0.0246**	**0.0491**
Activated carbon* time*concentration	**0.0851**	**0.0851**

Foot note:- Data given in the table are mean of three replication

Table 4.13 and Figure 4.21 (a-d) displayed that the adsorption capacity was decreased with the increased of activated carbon. It was noticed that at a low amount of activated carbon, high absorbency uptake was obtained. This result indicated that more surface area was made due to the increased mass of activated carbon. In addition to this, the table also revealed that among all the concentrations, absorbency was decreased at 1.0% activated carbon. From the statistical analysis, it indicated that all the factors and their interactions were found to be significantly influence at 5% level with respect to 2.5 N H_2SO_4 almond activated carbon.

The low absorbency was recorded in case of 2.5 N H_2SO_4 almond (filtered) activated carbon treated in different time periods at different concentrations. The lowest absorbency were recorded (0.680) at 0.1% concentration in 60 minute followed by (0.523) at 0.25% concentration in 90 minute, (0.500) at 0.5% concentration in 30 minute and 60 minute and (0.227) at 1.0% concentration in 60 minute of contact time period. In 2.5 N H_2SO_4 almond (ball milling) activated carbon, the lowest absorbency were recorded (0.670) in 90 minute at 0.1% concentration while the absorbency (0.600) in 90 minute at 0.25% concentration followed by (0.267) in 30 minute at 0.5% concentration and (0.147) in 30 minute and 60 minute of contact of time period at 1.0% concentration respectively. In case of commercial activated carbon the lowest absorbency were recorded (0.387) in 30 minute at 0.1% concentration followed by (0.150) in 30 minute and 90 minute at 0.25% concentration while the absorbency (0.007) in 90 minute at 0.5% concentration and (0.003) in 30 minute of contact time period at 1.0% concentration. In commercial activated carbon, the lowest value for the mean of concentration (1.0%) and the mean of activated carbon was recorded as 0.135 and 0.145 respectively. The lowest value for the mean of time was recorded as 0.365 at 90 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in ball milling (0.474).

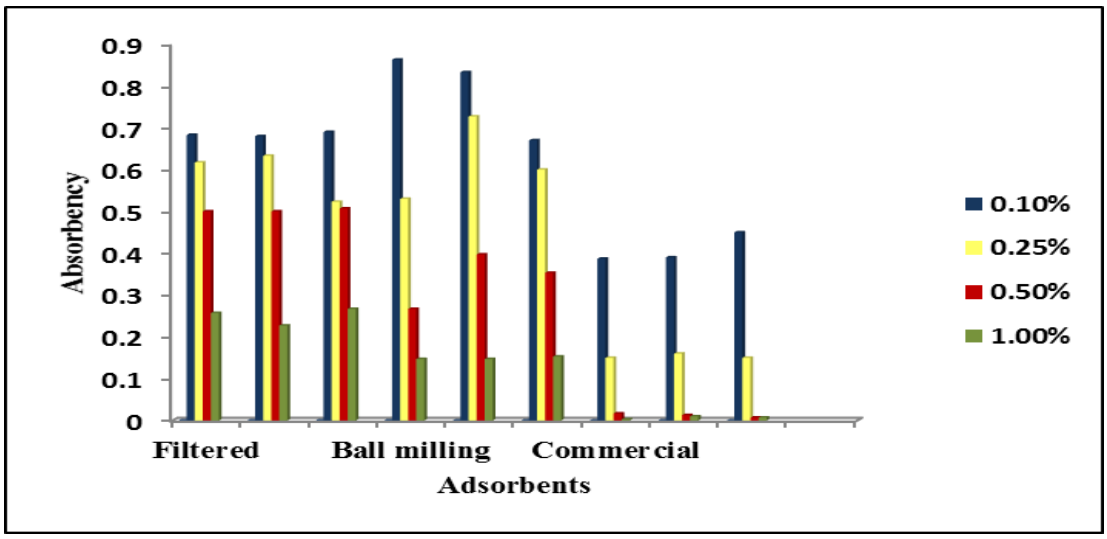


Figure 4.21 (a): Absorbency (2.5 N H_2SO_4 Almond) activated carbon

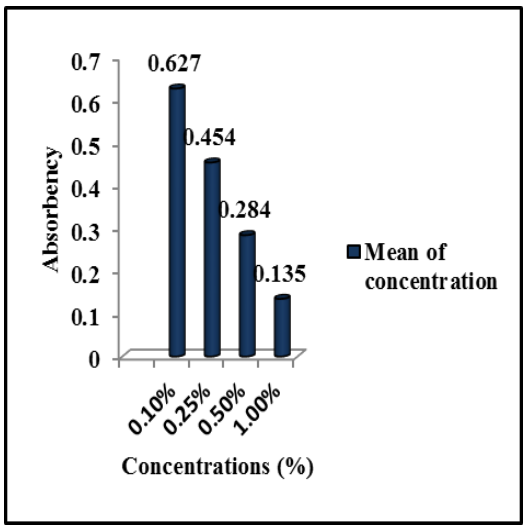


Figure 4.21 (b): Mean of concentration

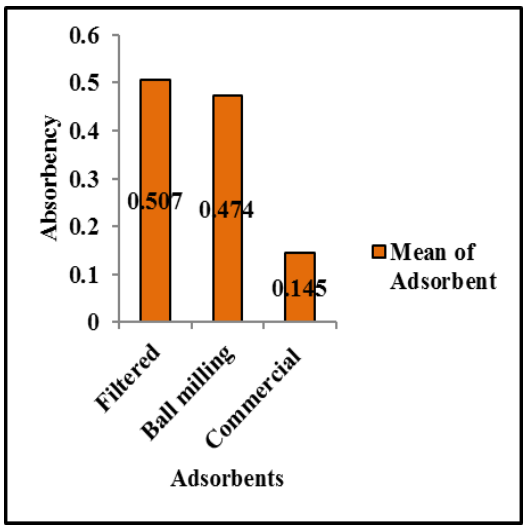


Figure 4.21 (c): Mean of activated carbon

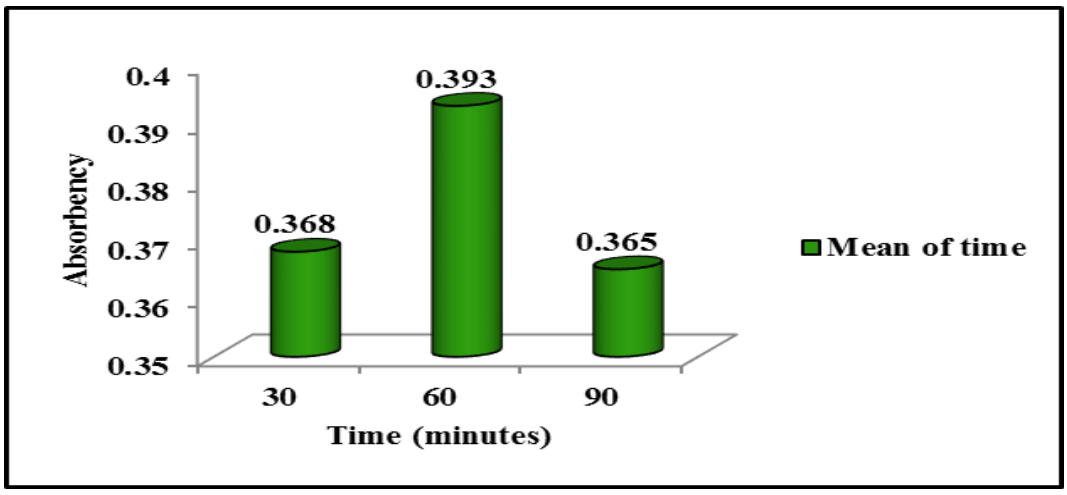


Figure 4.21 (d): Mean of time

Lamine, (2014) studied on the effectiveness of coffee grounds as an activated carbon support of phenol at room temperature and opined that the impregnation ratios have shown a strong influence on the capacity adsorption and surface area when the coffee residue was impregnated and then paralyzed at 600 and 700°C for 1 h. Tsai, *et al.*, 2001 have done a study on the Cleaner production of carbon activated carbons by utilizing agricultural waste corn cob and opined that the high adsorption capacities of activated carbons might be due to properties such as surface area, pore volume, and porosity.

Kebede and Gashaw (2017) have carried out a study on the removal of chromium and azo metal-complex dyes using activated carbon synthesized from tannery wastes and observed that with the dosage of increase in carbon there was an increase in removal efficiency, but adsorption capacity decreased and attains equilibrium which might be due to electrostatic interactions and interference of binding site which have an influence to reduce activated carbon densities (Ahmed Said *et al.*, 2013).

Plate 4.15 (a): 30 minute

Plate 4.15 (b): 60 minute

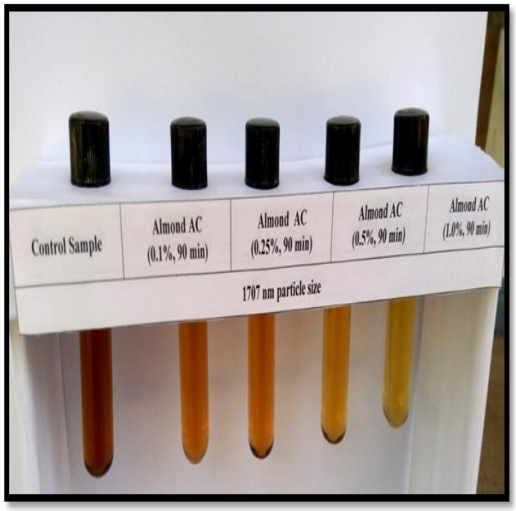


Plate 4.15 (c): 90 minute

PLATE 4.15: 2.5 N H_2SO_4 Almond (Filtered) activated carbons at different time

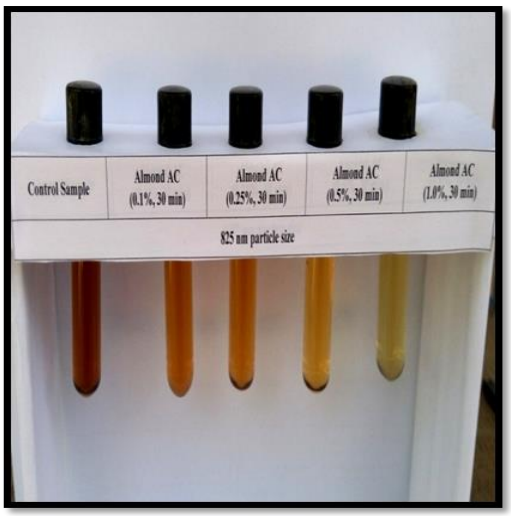


Plate 4.16 (a): 30 minute

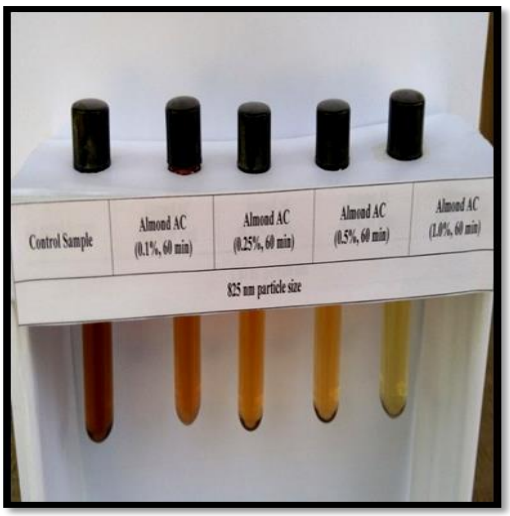


Plate 4.16 (b): 60 minute

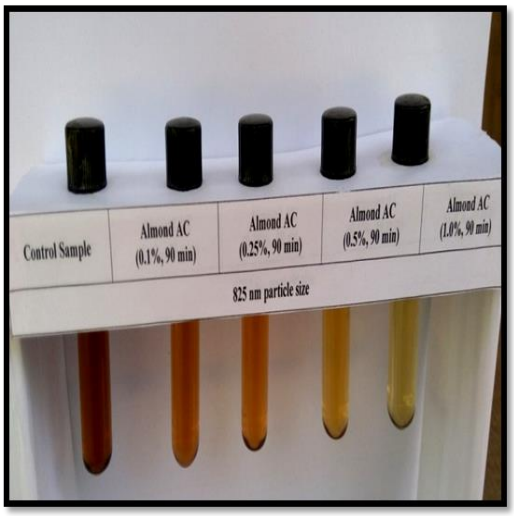


Plate 4.16 (c): 90 minute

PLATE 4.16: 2.5 N H_2SO_4 Almond (ball milling) activated carbons at different time

Plate 4.17 (a): 0.1% Concentration

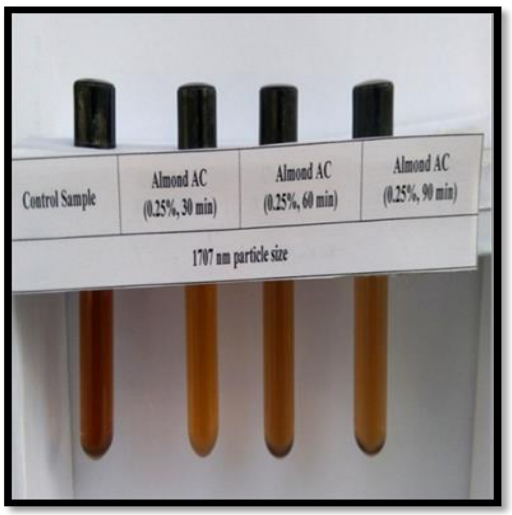


Plate 4.17 (b): 0.25% Concentration

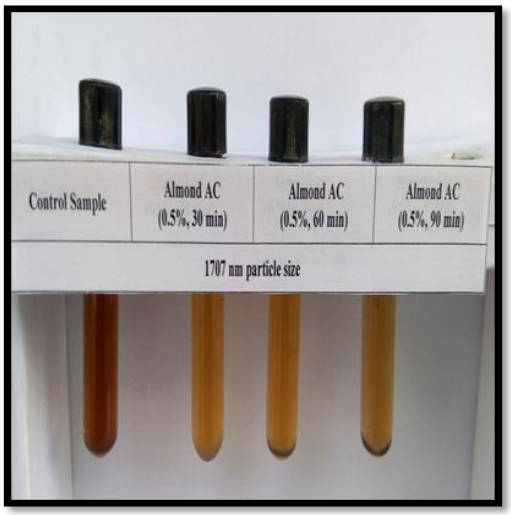


Plate 4.17 (c): 0.5% Concentration

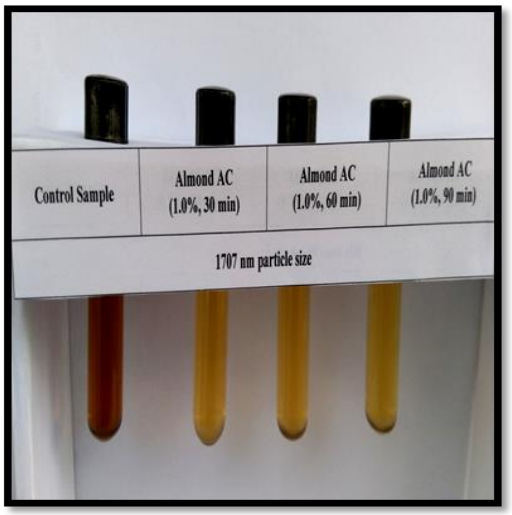


Plate 4.17 (d): 1.0% Concentration

PLATE 4.17: 2.5 N H_2SO_4 Almond (Filtered) activated carbons at different concentrations

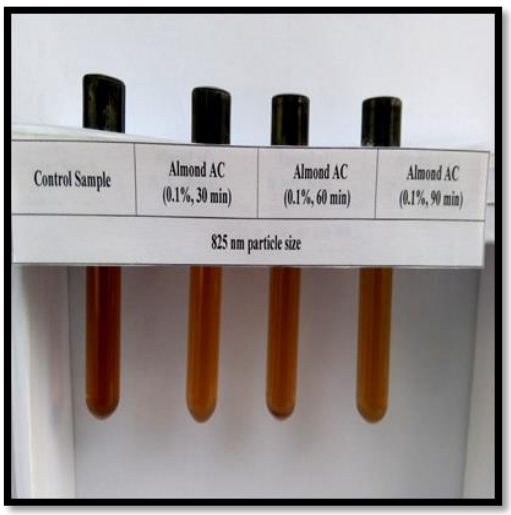


Plate 4.18 (a): 0.1% Concentration

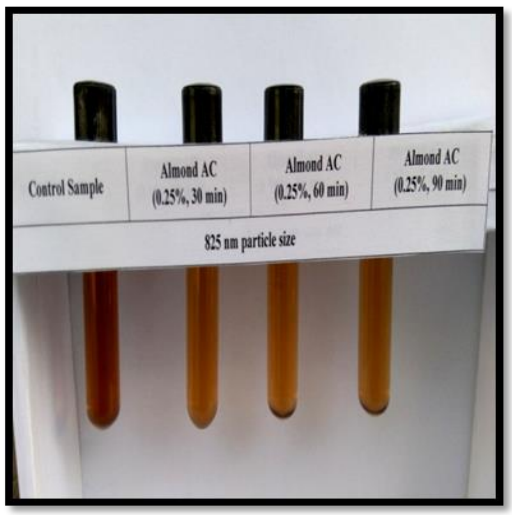


Plate 4.18 (b): 0.25% Concentration

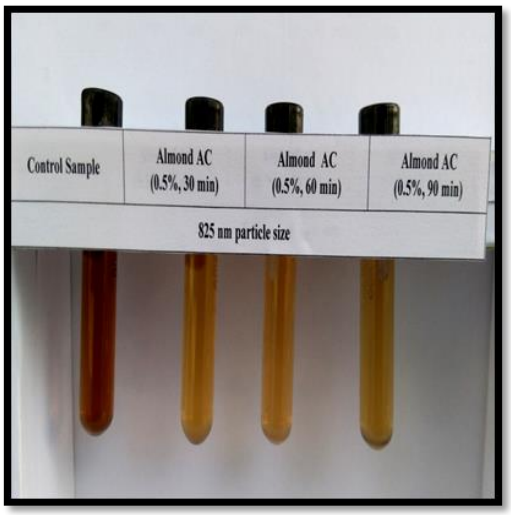


Plate 4.18 (c): 0.5% Concentration

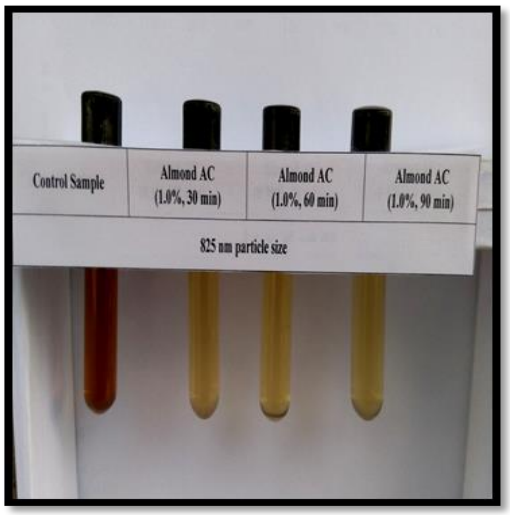


Plate 4.18 (d): 1.0% Concentration

PLATE 4.18: 2.5 N H_2SO_4 Almond (ball milling) activated carbons at different concentrations

4.10.2 Removal of Metal complex dye effluent using 2.5 N H_3PO_4 Rice Bran activated carbon

Table 4.14: Absorbency of 2.5 N H_3PO_4 Rice Bran activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.1%	0.510	0.470	0.453	0.533	0.553	0.570	0.387	0.390	0.450	**0.480**	30	**0.402**
0.25%	0.550	0.517	0.557	0.580	0.583	0.597	0.150	0.160	0.150	**0.427**	60	**0.387**
0.5%	0.633	0.580	0.600	0.487	0.497	0.497	0.017	0.013	0.007	**0.370**	90	**0.401**
1.0%	0.677	0.607	0.650	0.293	0.267	0.277	0.003	0.010	0.007	**0.310**		
Mean of Activated carbon	**0.567**			**0.478**			**0.145**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0104**	**0.0207**
Time	**0.0104**	**NS**
Concentration	**0.0120**	**0.0239**
Activated carbon*time	**0.0180**	**NS**
Activated carbon* concentration	**0.0208**	**0.0415**
Time*concentration	**0.0208**	**NS**
Activated carbon* time*concentration	**0.0360**	**NS**

Foot note:- Data given in the table are mean of three replication

Table 4.14 and figure 4.22 (a-d) displayed that the adsorption capacity was decreased with the increased of activated carbon. It was noticed that at a low amount of activated carbon, high absorbency uptake was obtained. This result indicated that more surface area was made due to the increased mass of activated carbon. In addition to this, the table also revealed that among all the concentrations, absorbency was decreased at 1.0% activated carbon. From the statistical analysis, it indicated that all the factors and their interactions except time, activated carbon*time, time*concentration and activated carbon* time*concentration were found to be non significant at 5% level with respect to 2.5 N H_3PO_4 rice bran activated carbon.

In 2.5 N H_3PO_4 rice bran (filtered) activated carbon, the lowest absorbency was recorded in a different time and different concentrations. The lowest absorbency was recorded (0.453) at 0.1% concentration in 90 minute followed by (0.517) at 0.25% concentration in 60 minute (0.580) at 0.5% concentration in 60 minute and (0.607) at 1.0% concentration in 60 minute of contact time period respectively. In 2.5 N H_3PO_4 rice bran (ball milling) activated carbon, the lowest absorbency was recorded (0.533) at 0.1% concentration in 30 minute, (0.580) at 0.25% concentration in 30 minute, (0.487) at 0.5% concentration in 30 minute and (0.267) at 1.0% concentration in 60 minute of contact time period respectively. In the commercial activated carbon, the lowest absorbency was recorded (0.387) in 30 minute at 0.1% concentration, (0.150) in 30 minute and 90 minute at 0.25% concentration, (0.007) in 90 minute at 0.5% concentration and (0.003) in 30 minute at 1.0% concentration. In commercial activated carbon, the lowest value for the mean of concentration (1.0%) and the mean of activated carbon was recorded as 0.310 and 0.145 respectively. The lowest value for mean of time was recorded as 0.387 at 60 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in ball milling (0.478).

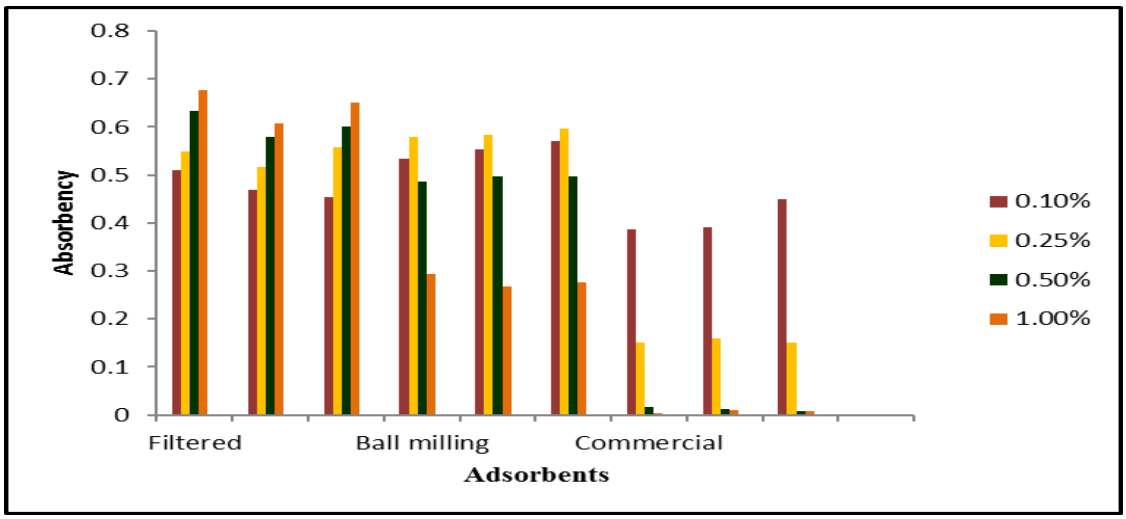


Figure 4.22 (a): Absorbency of (2.5 N H_3PO_4 Rice Bran) activated carbon

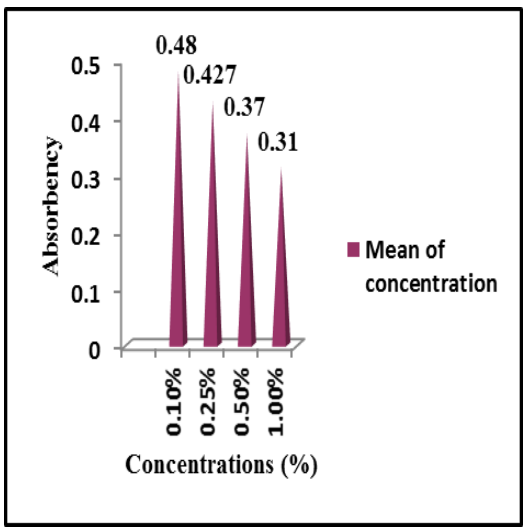


Figure 4.22 (b): Mean of concentration

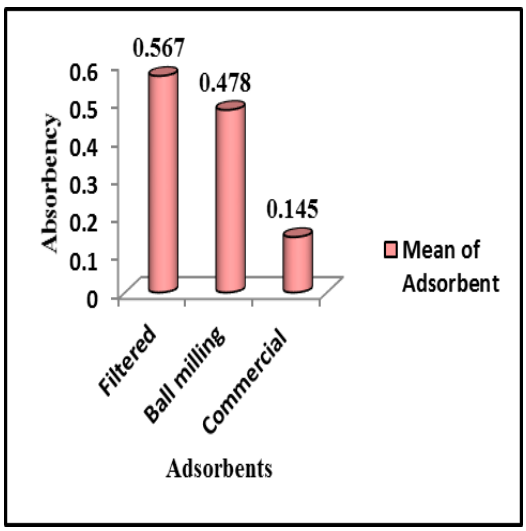


Figure 4.22 (c): Mean of activated carbon

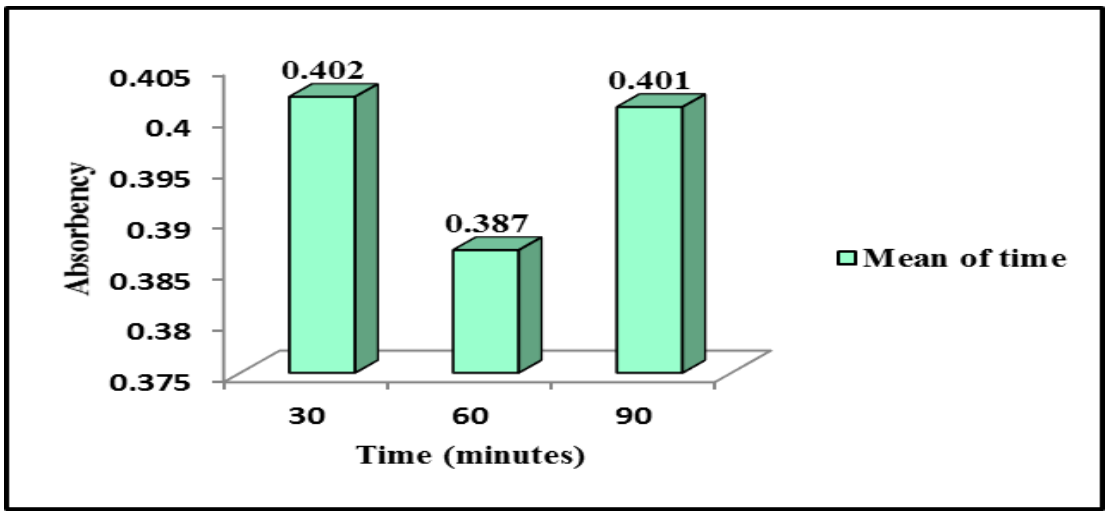


Figure 4.22 (d): Mean of time

Rao *et al.*, (2010) investigated on effect of sodium dodecyl sulfate (SDS) on the adsorption of Zn(II) and Ni(II) on carbon derived from mustard oil cake (CMOC) found that the percentage adsorption increases while adsorption capacity decreases as the doses of CMOC is increased which might be due to the fact that some adsorption sites may remain unsaturated during the adsorption process where as the number of sites available for adsorption increases by increasing the activated carbon doses and results in increasing removal efficiency of the activated carbon. These results are in agreement with Sharma and Foster (1993) on the removal of hexavalent chromium using sphagnum moss peat.

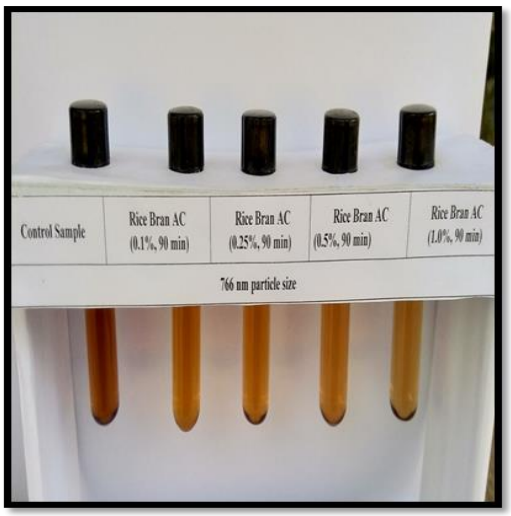


Plate 4.19 (a): 30 minute

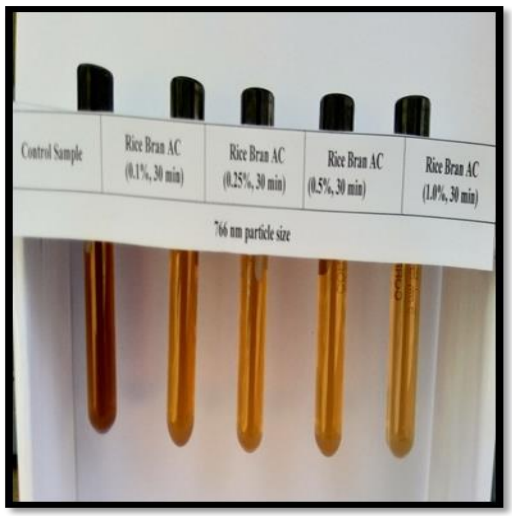


Plate 4.19 (b): 60 minute

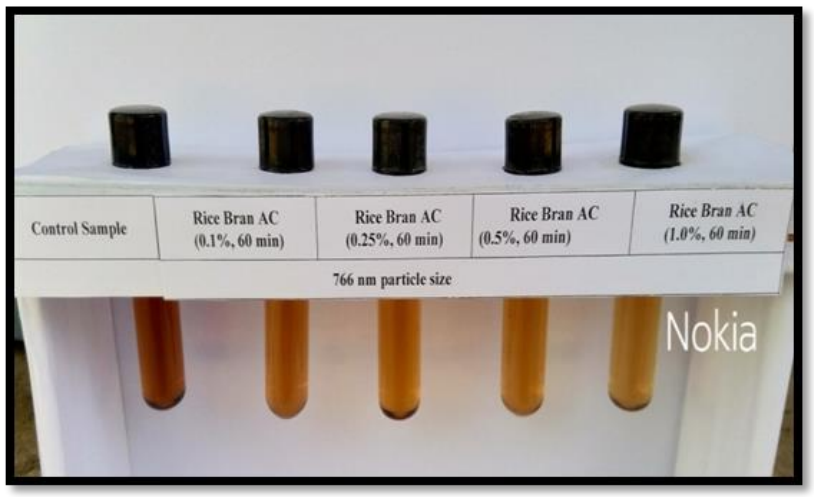


Plate 4.19 (c): 90 minute

PLATE 4.19: 2.5 N H_3PO_4 Rice Bran (Filtered) activated carbons at different time

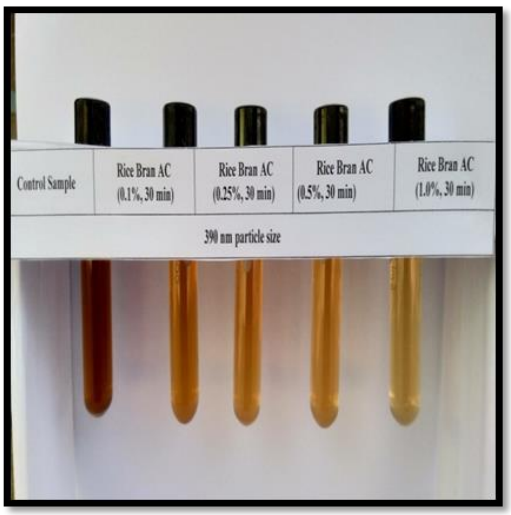


Plate 4.20 (a): 30 minute

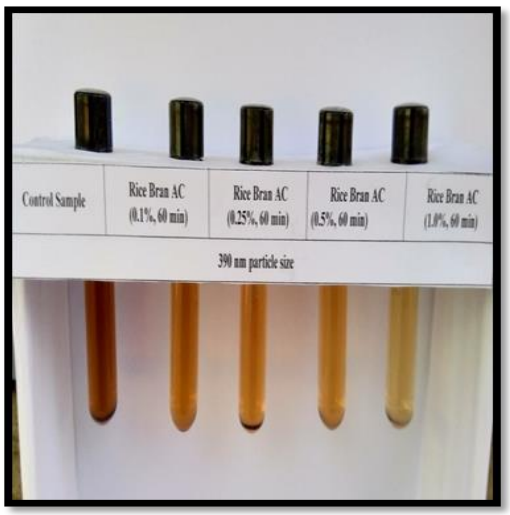


Plate 4.20 (b): 60 minute

Plate 4.20 (c): 90 minute

PLATE 4.20: 2.5 N H_3PO_4 Rice Bran (ball milling) activated carbons at different time

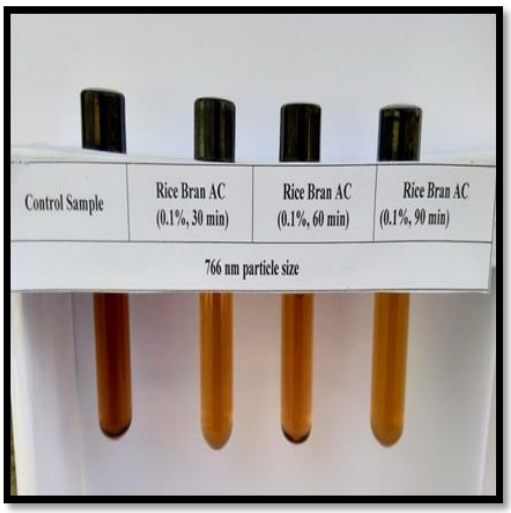


Plate 4.21 (a): 0.1% Concentration

Plate 4.21 (b): 0.25% Concentration

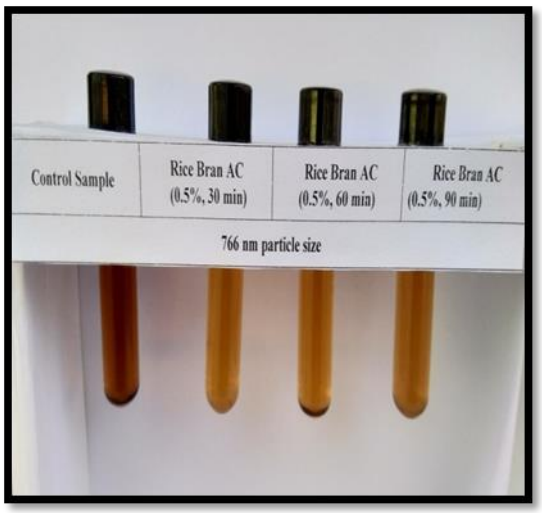


Plate 4.21 (c): 0.5% Concentration

Plate 4.21 (d): 1.0% Concentration

PLATE 4.21: 2.5 N H_3PO_4 Rice Bran (Filtered) activated carbons at different concentrations

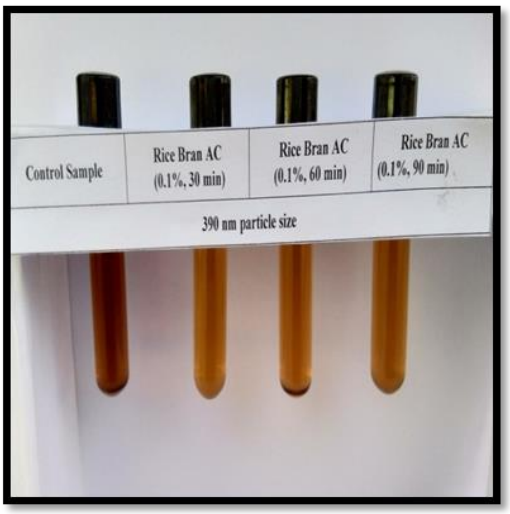


Plate 4.22 (a): 0.1% Concentration

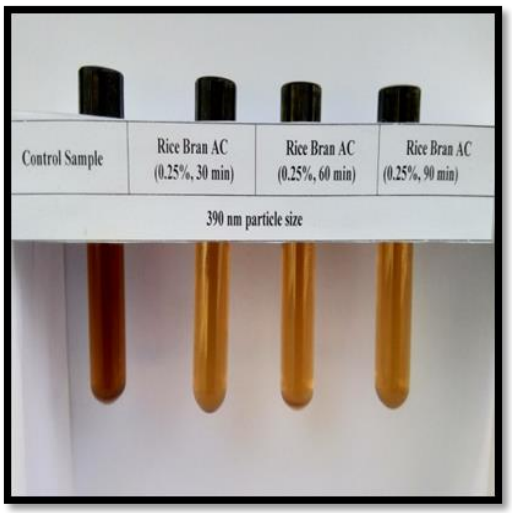


Plate 4.22 (b): 0.25% Concentration

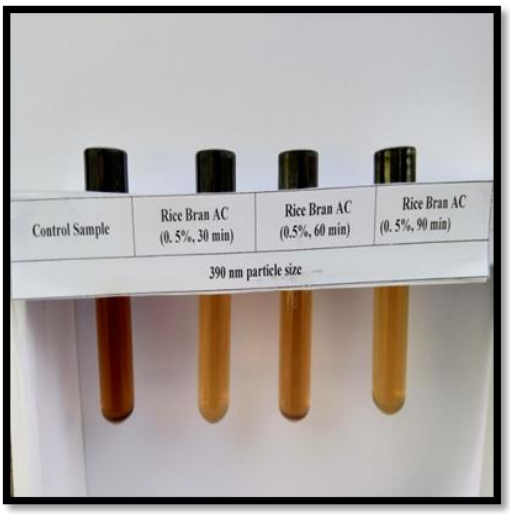


Plate 4.22(c): 0.5% Concentration

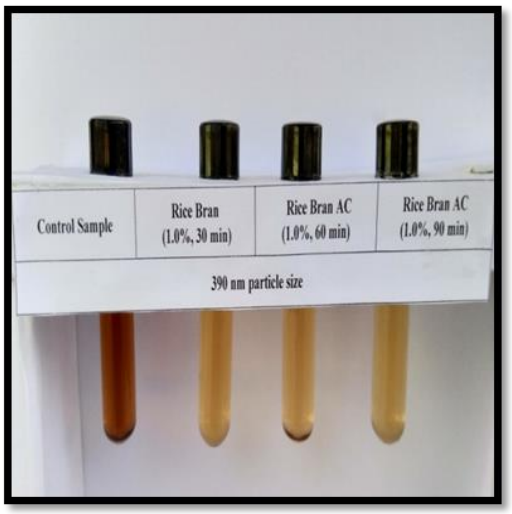


Plate 4.22 (d): 1.0% Concentration

PLATE 4.22: 2.5 N H_3PO_4 Rice Bran (ball milling) activated carbons at different concentrations

4.10.3 Removal of Metal complex dye effluent using 2.5 N H_2SO_4 Sesame activated carbon

Table 4.15: Absorbency of 2.5 N H_2SO_4 Sesame activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.1%	0.490	0.543	0.523	0.630	0.717	0.620	0.387	0.390	0.450	**0.528**	30	**0.330**
0.25%	0.467	0.503	0.510	0.570	0.557	0.570	0.150	0.160	0.150	**0.404**	60	**0.342**
0.5%	0.437	0.363	0.430	0.357	0.373	0.373	0.017	0.013	0.007	**0.263**	90	**0.346**
1.0%	0.243	0.243	0.280	0.213	0.227	0.230	0.003	0.010	0.007	**0.162**		
Mean of Activated carbon	**0.419**			**0.453**			**0.145**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0065**	**0.0129**
Time	**0.0065**	**NS**
Concentration	**0.0075**	**0.0149**
Activated carbon*time	**0.0112**	**NS**
Activated carbon* concentration	**0.0130**	**0.0259**
Time*concentration	**0.0130**	**0.0259**
Activated carbon* time*concentration	**0.0225**	**0.0448**

Foot note:- Data given in the table are mean of three replication

Table 4.15 and figure 4.23 (a-d) illustrated that the adsorption capacity was decreased with the increased of activated carbon. It was noticed that at a low amount of activated carbon, high absorbency uptake was obtained. This result indicated that more surface area was made due to the increased mass of activated carbon. In addition to this, the table also revealed that among all the concentrations, absorbency was decreased at 1.0% activated carbon. From the statistical analysis, it indicated that all the factors and their interactions except time and activated carbon*time were found to be non significant at 5% level with respect to 2.5 N H_2SO_4 sesame activated carbon.

In 2.5 N H_2SO_4 sesame (filtered) activated carbon, the lowest absorbency was recorded in different time and different concentrations. The lowest absorbency were recorded (0.490) at 0.1% concentration in 30 minute followed by (0.467) at 0.25% concentration in 30 minute, (0.363) at 0.5% concentration in 60 minute and (0.243) at 1.0% concentration in 30 minute and 60 minute respectively. In 2.5 N H_2SO_4 sesame (ball milling) activated carbon, the lowest absorbency were recorded (0.620) at 0.1%concentration in 90 minute, (0.557) at 0.25% concentration in 60 minute, (0.357) at 0.5% concentration in 30 minute and (0.213) at 1.0% concentration in 30 minute of contact time period respectively. In commercial activated carbon, the lowest absorbency were recorded (0.387) in 30 minute at 0.1% concentration, (0.150) in 30 minute and 90 minute at 0.25% concentration, (0.007) in 90 minute at 0.5% concentration and (0.003) in 30 minute of contact time at 1.0% concentration. In commercial activated carbon, the lowest value for the mean of concentration (1.0%) and the mean of activated carbon was recorded as 0.162 and 0.145 respectively. The lowest value for the mean of time was recorded as 0.330 at 30 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in filtered (0.419).

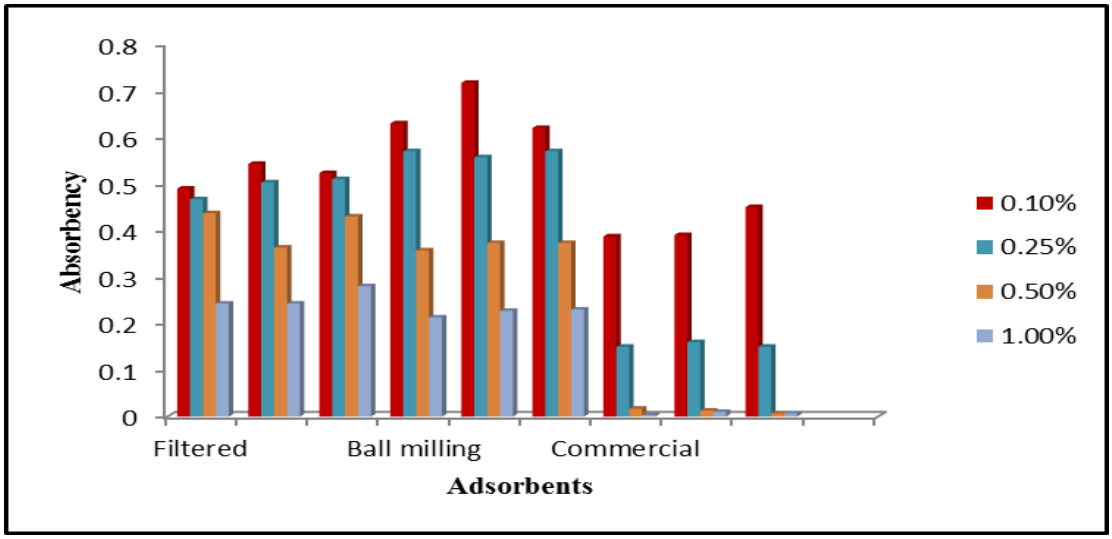


Figure 4.23 (a): Absorbency of (2.5 N H_2SO_4 Sesame) activated carbon

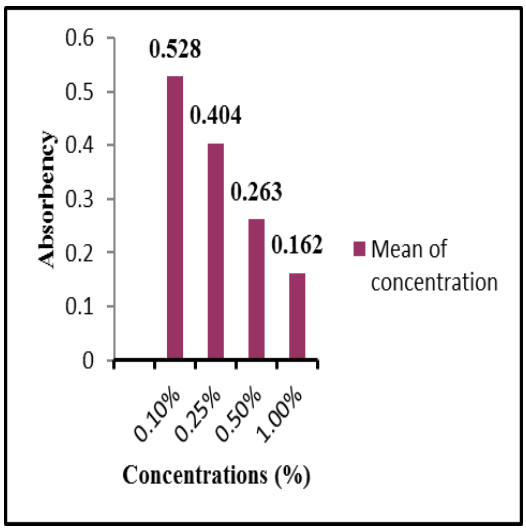


Figure 4.23 (b): Mean of concentration

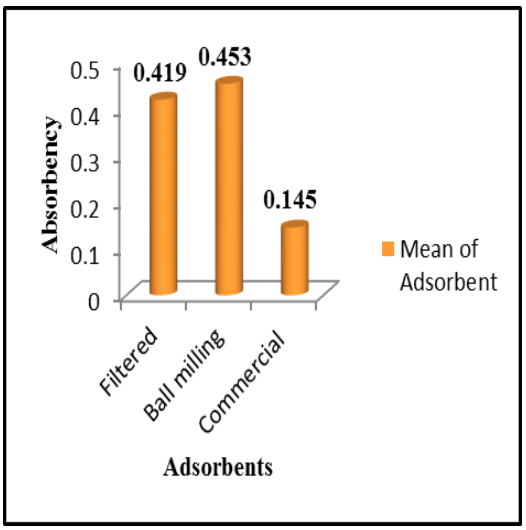


Figure 4.23 (c): Mean of activated carbon

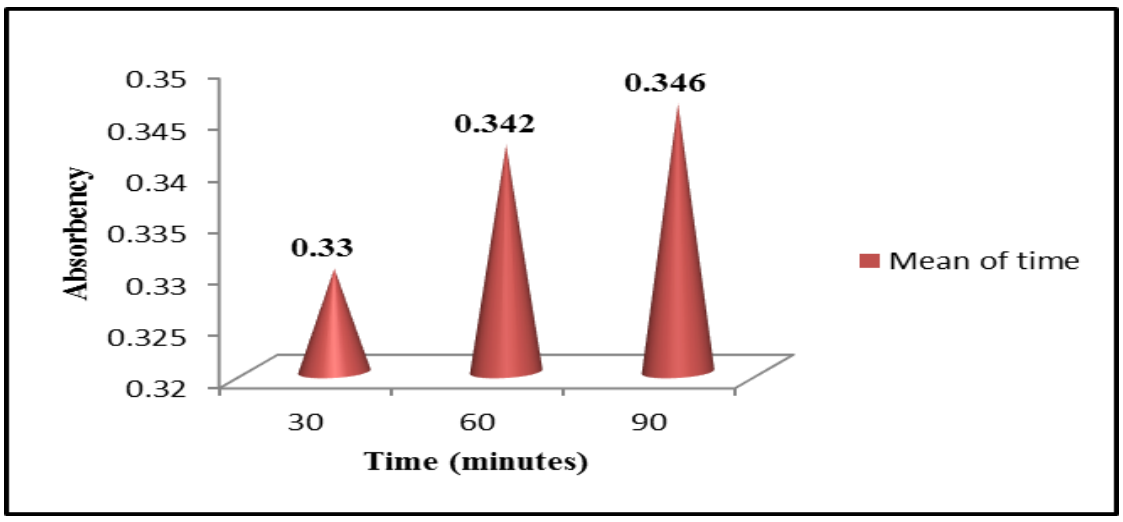


Figure 4.23 (d): Mean of time

Gottipati (2012) worked on preparation and characterization of microporous activated carbon from biomass and its application in the removal of chromium (vi) from aqueous phase found that the adsorption capacities rapidly decreased with increase of activated carbon doses which might be due to the fact that all the activated carbons have a limited number of active sites that would have achieved saturation above a certain adsorbate concentration. On the other hand it was found that there was gradual increase in the removal of Cr (VI) with an increase in activated carbon dosages because as the activated carbon doses increases the number of activated carbon particles increases and hence more Cr (VI) ions attach to the adsorption sites (Barkat *et al.*, 2009; Dubey and Gopal, 2007; Mohanty *et al.*, 2005; Ranganathan, 2000).

Plate 4.23 (a): 30 minute

Plate 4.23 (b): 60 minute

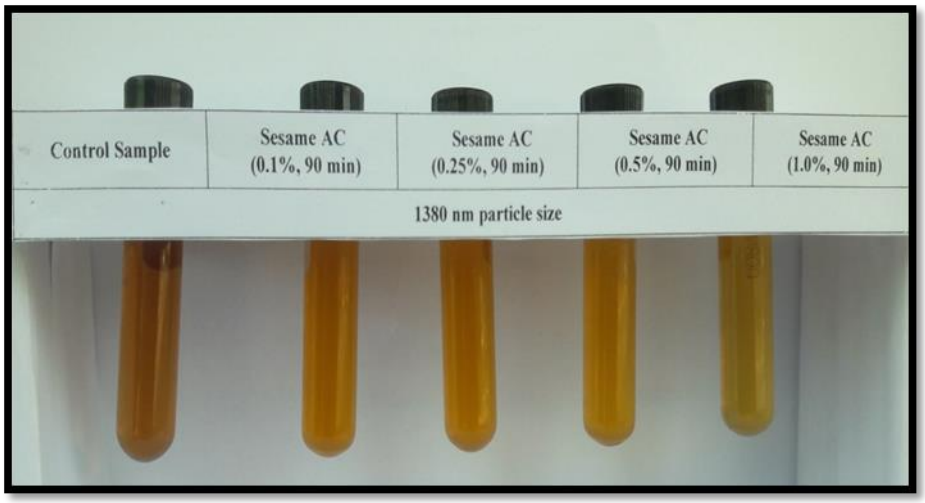


Plate 4.23 (c): 90 minute

PLATE 4.23: 2.5 N H_2SO_4 Sesame (Filtered) activated carbons at different time

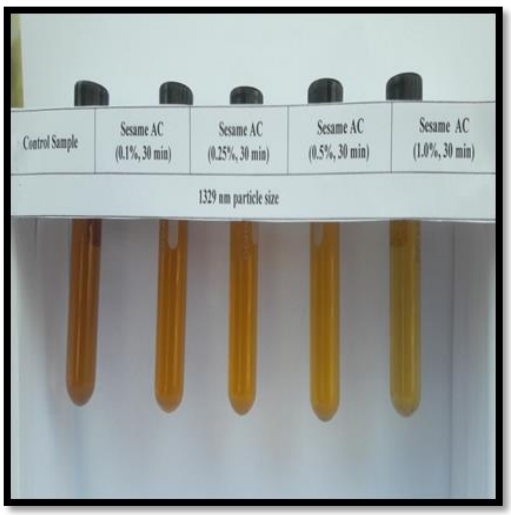


Plate 4.24 (a): 30 minute

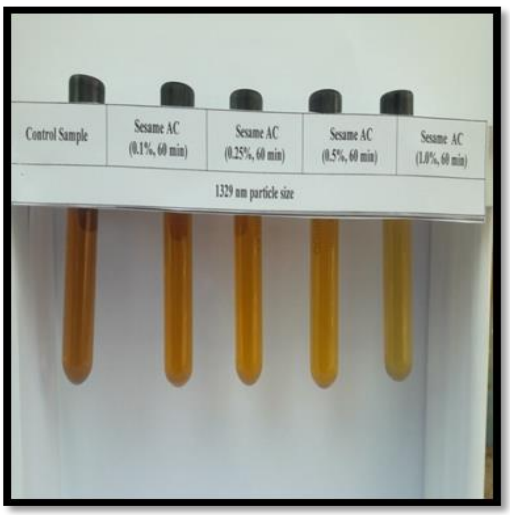


Plate 4.24 (b): 60 minute

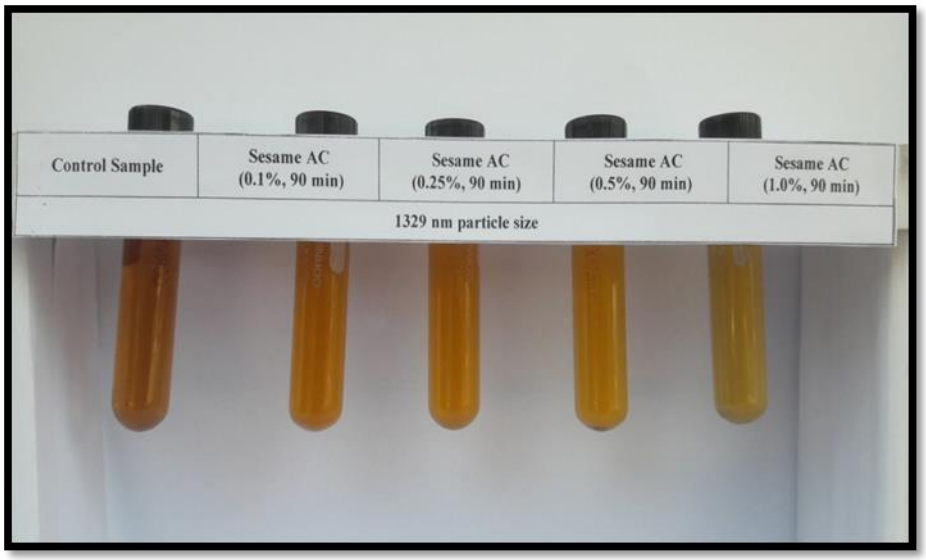


Plate 4.24 (c): 90 minute

PLATE 4.24: 2.5 N H_2SO_4 Sesame (ball milling) activated carbons at different time

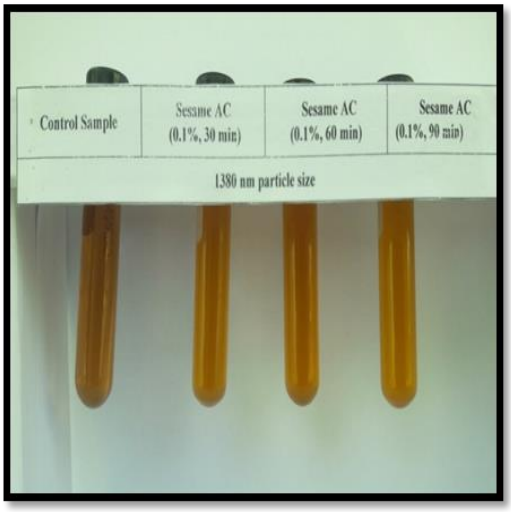


Plate 4.25 (a): 0.1% Concentration

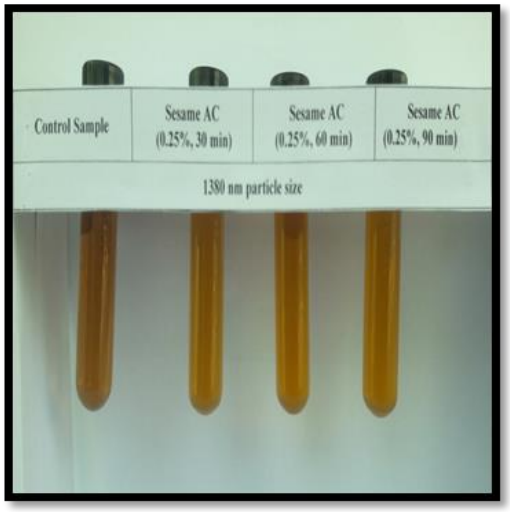


Plate 4.25 (b): 0.25% Concentration

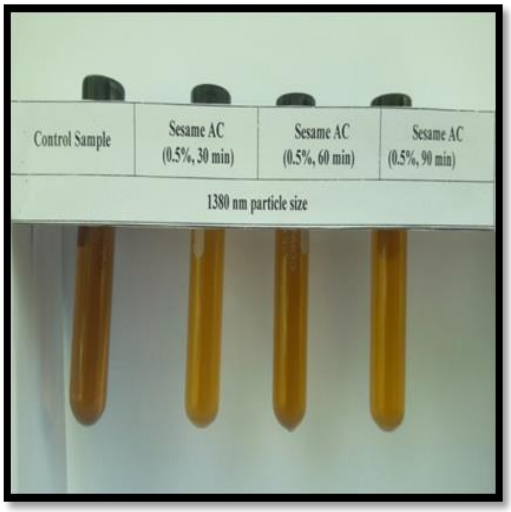


Plate 4.25 (c): 0.5% Concentration

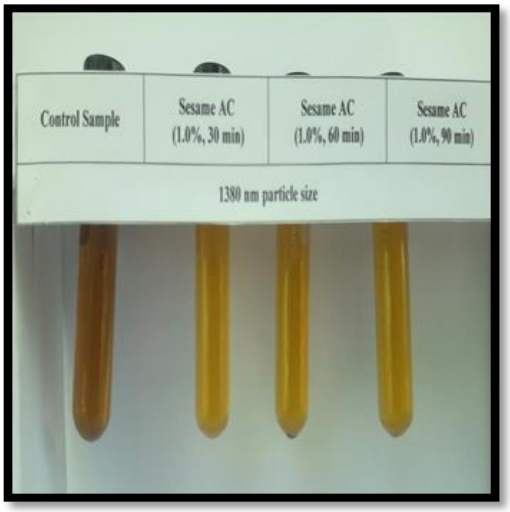


Plate 4.25 (d): 1.0% Concentration

PLATE 4.25: 2.5 N H_2SO_4 Sesame (Filtered) activated carbons at different concentrations

Plate 4.26 (a): 0.1% Concentration

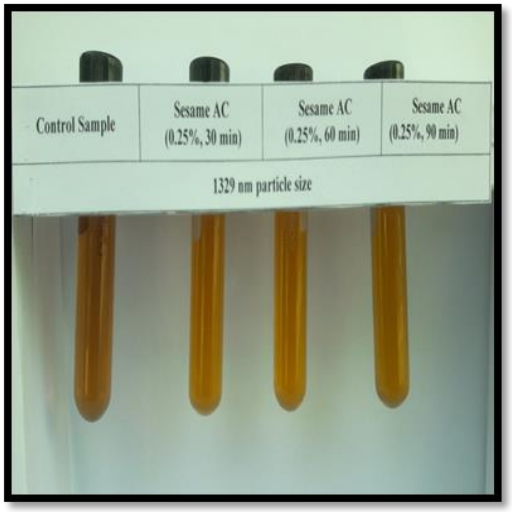


Plate 4.26 (b): 0.25% Concentration

Plate 4.26 (c): 0.5% Concentration

Plate 4.26 (d): 1.0% Concentration

PLATE 4.26: 2.5 N H_2SO_4 Sesame (ball milling) activated carbons at different concentrations

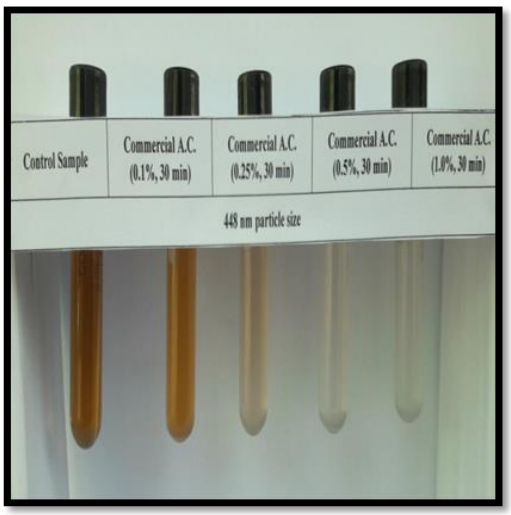


Plate 4.27(a): 30 minute

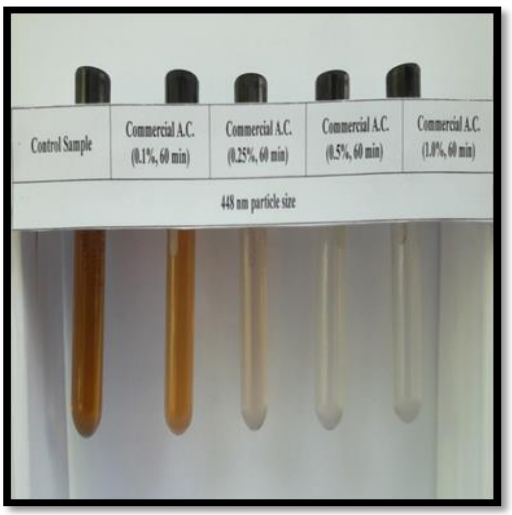


Plate 4.27 (b): 60 minute

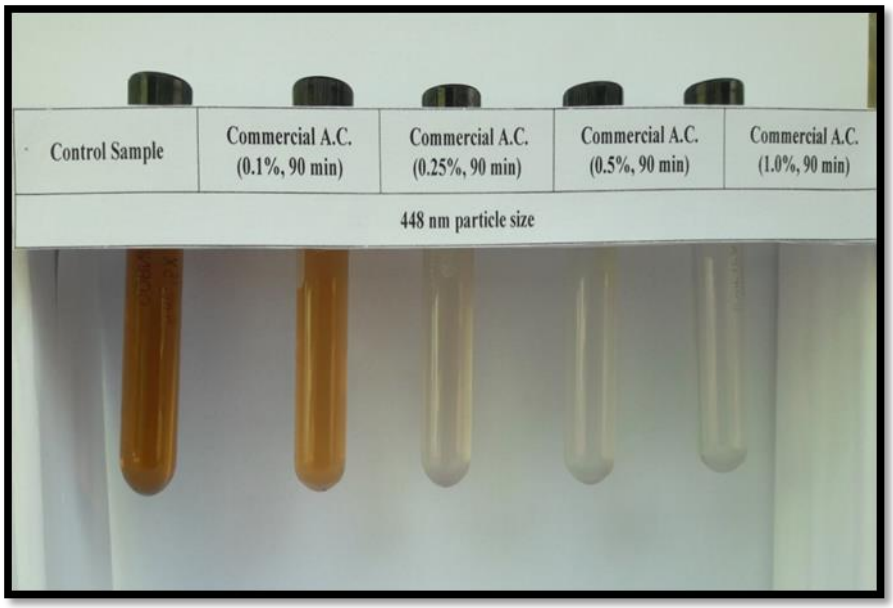


Plate 4.27 (c): 90 minute

PLATE 4.27: Commercial activated carbon at different time

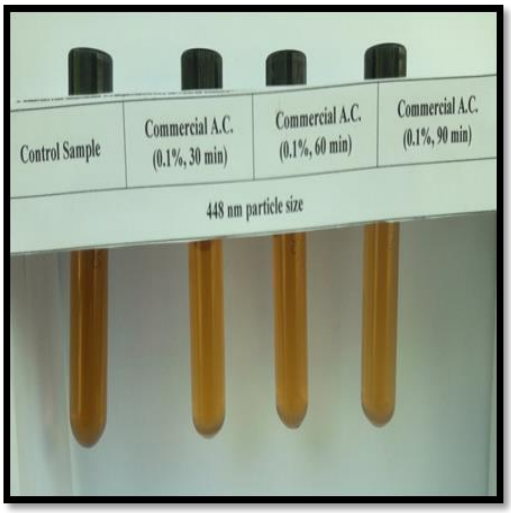


Plate 4.28 (a): 0.1% Concentration

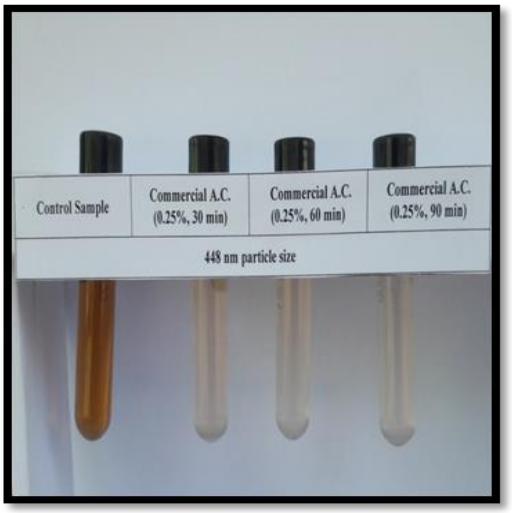


Plate 4.28 (b): 0.25% Concentration

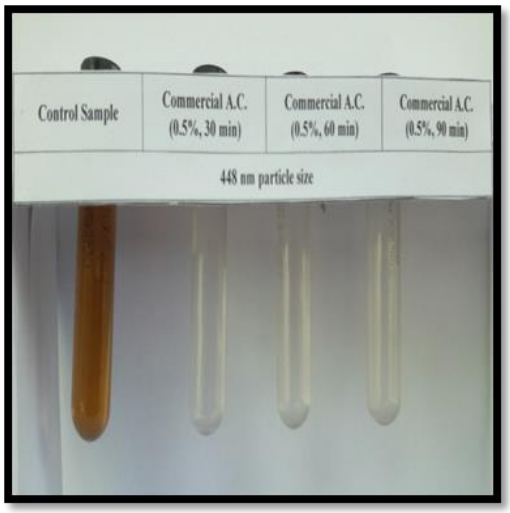


Plate 4.28 (c): 0.5% Concentration

Plate 4.28 (d): 1.0% Concentration

PLATE 4.28: Commercial activated carbon at different concentrations

4.11 pH of the effluent acid dye

4.11.1 pH of the effluent acid dye using 2.5 N H_3PO_4 Almond activated carbon

Table 4.16: pH of the effluent 2.5 N H_3PO_4 Almond activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.5%	5.110	5.130	5.153	5.333	5.327	5.120	7.200	7.233	7.187	**5.866**	30	**5.600**
1.0%	4.503	4.810	4.853	4.920	4.820	4.843	7.130	7.273	7.210	**5.596**	60	**5.645**
1.5%	4.713	4.733	4.703	4.753	4.733	4.613	7.247	7.267	7.287	**5.561**	90	**5.616**
2.0%	4.420	4.563	4.650	4.580	4.523	4.543	7.293	7.323	7.233	**5.459**		
Mean of Activated carbon	**4.779**			**4.843**			**7.240**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0161**	**0.0321**
Time	**0.0161**	**0.0321**
Concentration	**0.0186**	**0.0371**
Activated carbon*time	**0.0279**	**0.0557**
Activated carbon* concentration	**0.0322**	**0.0643**
Time*concentration	**0.0322**	**0.0643**
Activated carbon* time*concentration	**0.0558**	**0.1113**

Foot note:- Data given in the table are mean of three replication

Table 4.16 and figure 4.24 (a-d) illustrated that the pH was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, pH was decreased at 2.0% activated carbon. From the statistical analysis, it indicated that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 almond activated carbon.

The lowest pH recorded in 2.5 N H_3PO_4 almond (filtered) activated carbon were (4.42) at 2.0 % concentration in 30 minute followed by (4.703) at 1.5% concentration in 90 minute, (4.503) at 1.0% concentration in 30 minute and (5.11) at 0.5% concentration in 30 minute. In 2.5 N H_3PO_4 almond (ball milling) activated carbon, it was recorded (4.523) at 2.0% concentration in 60 minute followed by (4.613) at 1.5% concentration in 90 minute while the pH (4.820) at 1.0% concentration in 60 minute and (5.120) at 0.5% concentration in 90 minute of contact time period respectively. In the commercial activated carbon, pH were recorded (7.233) at 2.0% concentration in 90 minute, (7.247) at 1.5% concentration in 30 minute, (7.130) 1.0% concentration in 30 minute and (7.187) at 0.5% concentration in 90 minute respectively. The lowest value for the pH mean of concentration (2.0%) and the mean of activated carbon were recorded as 5.459 and 7.240 respectively in commercial activated carbon. The lowest value for the pH mean of time was recorded as 5.600 at 30 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the pH mean of activated carbon was recorded in filtered (4.779).

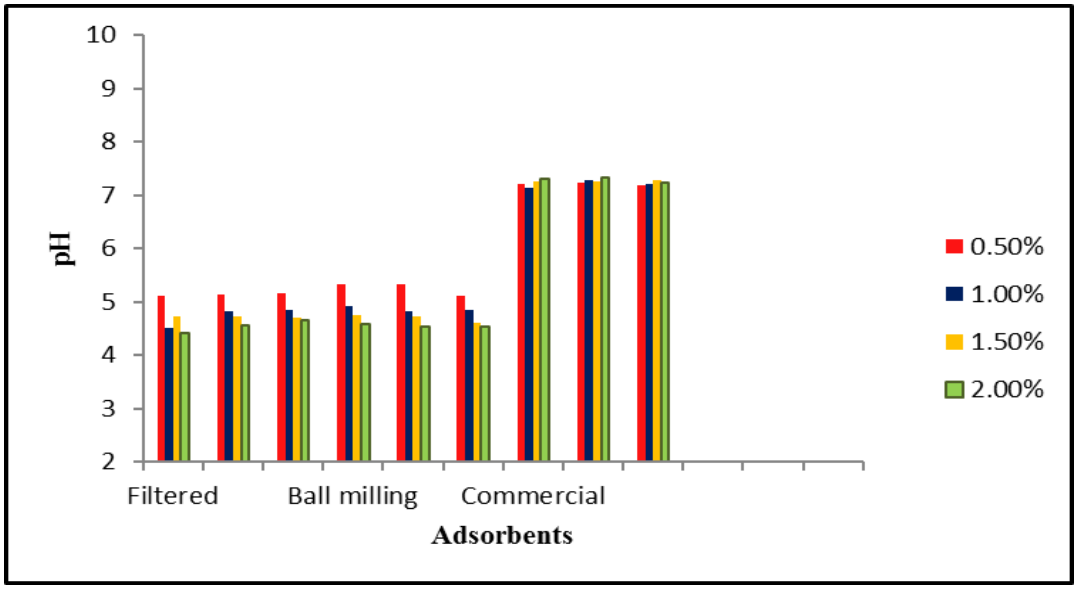
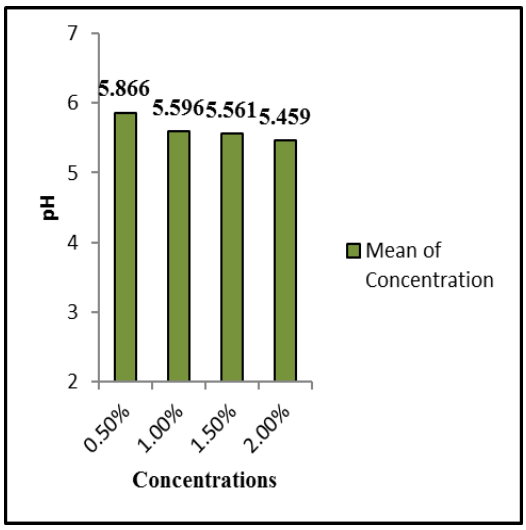


Figure 4.24(a): pH of the effluent (2.5 N H_3PO_4 Almond) activated carbon



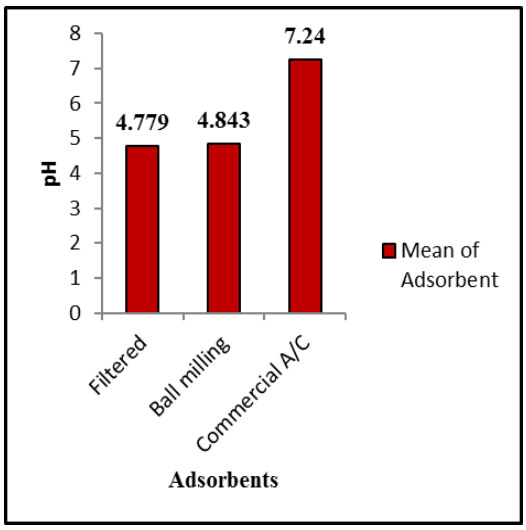


Figure 4.24 (b): Mean of concentration **Figure 4.24 (c): Mean of activated carbon**

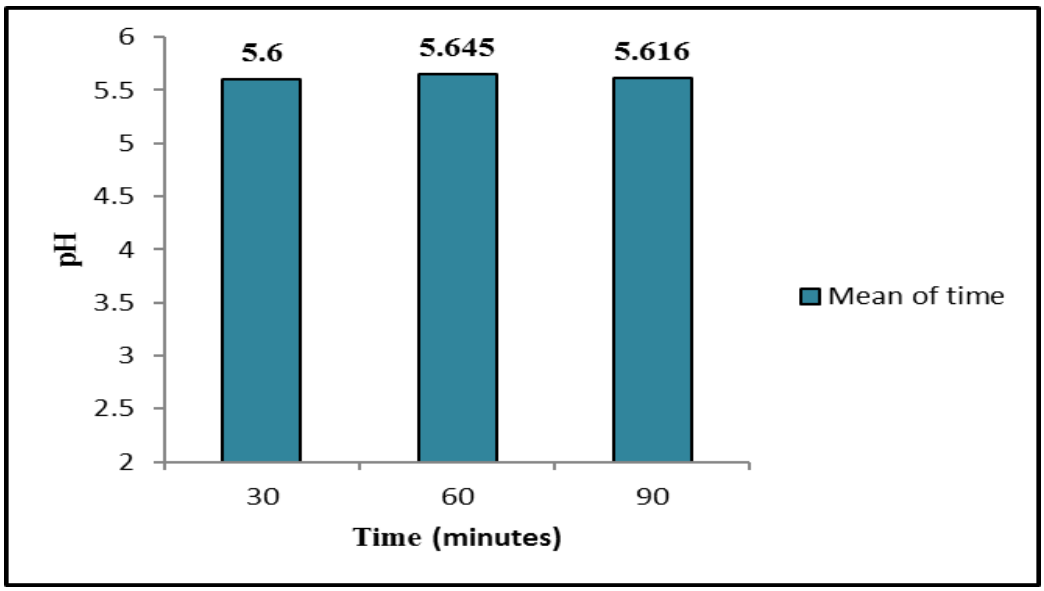


Figure 4.24 (d): Mean of time

Akpen *et al.,* (2017) studied activated carbon from a local variety of mango (*Magnifera indica)* by chemical activation with Zinc Chloride ($ZnCl_2$) before and after carbonization found that the effect of pH at 7 on the removal of color by the MSEAC was achieved 100% for commercial activated carbons. Okiemmen *et al.* (2007) reported that for most adsorption applications, carbons with pH 6-8 are acceptable.

4.11.2 pH of the effluent acid dye using 1 N H_3PO_4 Coconut activated carbon

Table 4.17: pH of the effluent 1 N H_3PO_4 Coconut activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.5%	6.027	5.737	6.323	5.333	5.330	5.347	7.200	7.233	7.187	**6.191**	30	**5.957**
1.0%	5.527	5.433	5.260	5.247	5.100	5.120	7.130	7.273	7.210	**5.922**	60	**5.897**
1.5%	5.260	5.317	5.137	5.150	4.957	4.893	7.247	7.267	7.287	**5.835**	90	**5.908**
2.0%	4.940	4.943	4.940	5.130	4.853	4.960	7.293	7.323	7.233	**5.735**		
Mean of Activated carbon	**5.404**			**5.118**			**7.240**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0081**	**0.0161**
Time	**0.0081**	**0.0161**
Concentration	**0.0093**	**0.0186**
Activated carbon*time	**0.0140**	**0.0279**
Activated carbon* concentration	**0.0162**	**0.0323**
Time*concentration	**0.0162**	**0.0323**
Activated carbon* time*concentration	**0.0280**	**0.0559**

Foot note:- Data given in the table are mean of three replication

Table 4.17 and figure 4.25 (a-d) illustrated that the pH was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, pH was decreased at 2.0% activated carbon. From the statistical analysis, it indicated that all the factors and their interactions were found to be significant at 5% level with respect to 1 N H_3PO_4 coconut activated carbon.

The lowest pH was recorded in 1 N H_3PO_4 coconut (filtered) activated carbon were (4.940) at 2.0% concentration in 30 minute and 90 minute (5.137) at 1.5% concentration in 90 minute, (5.260) at 1.0% concentration in 90 and (5.737) at 0.5% concentration in 60 minute of contact time. In 1 N H_3PO_4 coconut (ball milling) activated carbon, it was recorded (4.853), at 2.0% concentration in 60 minute, (4.893) at 1.5% concentration in 90 minute, (5.100) at 1.0% concentration in 60 minute and (5.330) at 0.5% concentration in 60 minute contact of time period respectively. In the commercial activated carbon, pH were recorded (7.233) at 2.0% concentration in 90 minute followed by (7.247) at 1.5% concentration in 30 minute while the pH (7.130) at 1.0% concentration in 30 minute and (7.187) at 0.5% concentration in 90 minute contact of time respectively. The lowest value for the pH mean of concentration (2.0%) and the mean of activated carbon was recorded as 5.735 and 7.240 respectively in commercial activated carbon. The lowest value for the pH mean of time was recorded as 5.897 at 60 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the pH mean of activated carbon was recorded in ball milling (5.118).

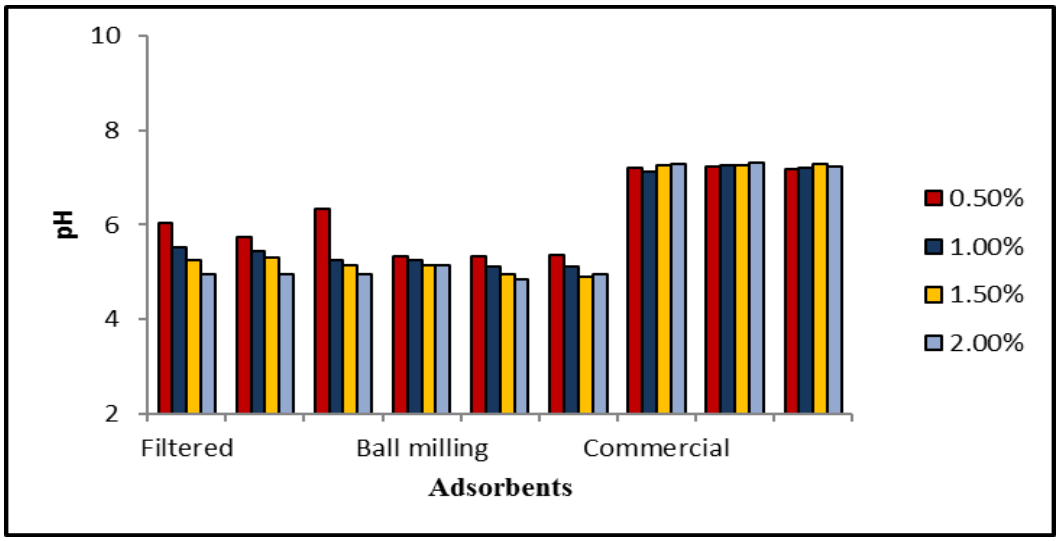
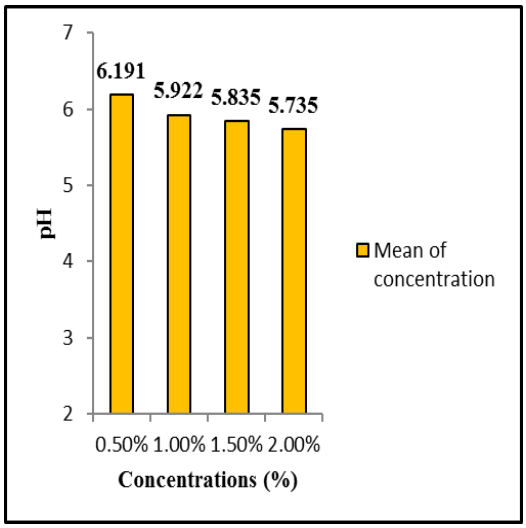


Figure 4.25 (a): pH of the effluent (1 N H_3PO_4 Coconut) activated carbon



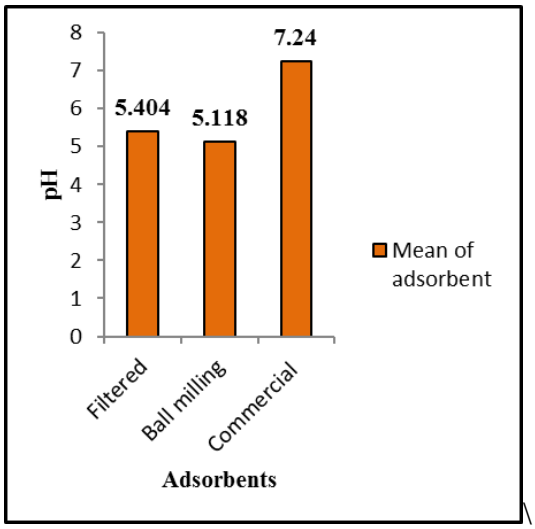


Figure 4.25 (b): Mean of concentration **Figure 4.25 (c): Mean of activated carbon**

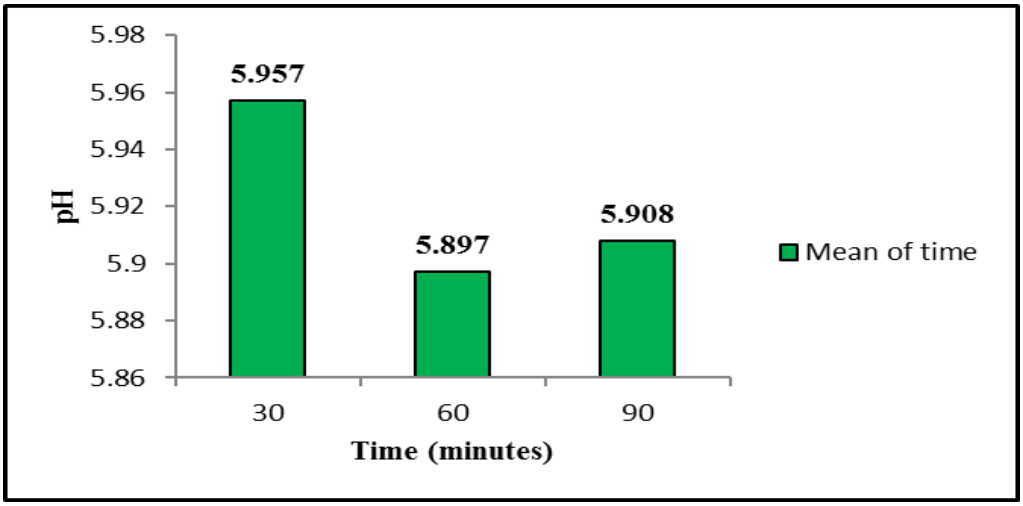


Figure 4.25 (d): Mean of time

Baskaralingam *et al.*, (2006) studied on adsorption of acid dye onto organobentonite found that acid red151 (AR 151) showed minimum and maximum pH at 3 and 7 respectively, which might be due to the electrostatic attraction between the negatively charged activated carbon and positively charged activated carbon and the chemical reaction between the activated carbon. At a lower pH, more protons will be available, thereby increasing the electrostatic attraction between the negatively charged SO^{3-} anion and the positively charged adsorption site. As the pH of the solution increases, the positive charge on the surface decreases, and the number of negatively charged sites increases.

4.11.3 pH of the effluent acid dye using 2.5 N H_3PO_4 Mustard activated carbon

Table 4.18: pH of the effluent 2.5 N H_3PO_4 Mustard activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.5%	5.177	5.263	5.037	5.160	5.203	5.137	7.200	7.233	7.187	**5.844**	30	**5.676**
1.0%	5.117	4.973	4.843	4.877	4.863	4.930	7.130	7.273	7.210	**5.691**	60	**5.668**
1.5%	4.887	4.703	4.657	4.670	4.727	4.730	7.247	7.267	7.287	**5.575**	90	**5.609**
2.0%	4.690	4.593	4.533	4.667	4.597	4.530	7.293	7.323	7.233	**5.496**		
Mean of Activated carbon	**4.873**			**4.841**			**7.240**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0034**	**0.0067**
Time	**0.0034**	**0.0067**
Concentration	**0.0039**	**0.0077**
Activated carbon*time	**0.0058**	**0.0116**
Activated carbon* concentration	**0.0067**	**0.0134**
Time*concentration	**0.0067**	**0.0134**
Activated carbon* time*concentration	**0.0116**	**0.0232**

Foot note:- Data given in the table are mean of three replication

Table 4.18 and figure 4.26 (a-d) showed that the pH was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, pH was decreased at 2.0% activated carbon. From the statistical analysis, it indicated that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 mustard activated carbon.

In 2.5 N H_3PO_4 mustard (filtered) activated carbon, pH were recorded (4.533) at 2.0% concentration, (4.657) at 1.5% concentration, (4.843) at 1.0% concentration and (5.037) at 0.5% concentration in 90 minutes of contact time period respectively. In 2.5 N H_3PO_4 mustard (ball milling) activated carbon, pH were recorded (4.530) at 2.0% concentration in 90 minute, (4.670) at 1.5% concentration in 30 minute, (4.863) at 1.0% concentration in 60 minute and (5.137) at 0.5% in 90 minutes of contact time period respectively. In case the commercial activated carbon, pH, were recorded (7.233) in 90 minute at 2.0% concentration followed by (7.247) in 30 minute at 1.5% concentration while the pH (7.130) in 30 minute at 1.0% concentration and (7.187) in 90 minute of contact time period at 0.5% concentration respectively. The lowest value for the pH mean of concentration (2.0%) and the mean of activated carbon was recorded as 5.496 and 7.240 respectively in commercial activated carbon. The lowest value for the pH mean of time was recorded as 5.609 at 90 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the pH mean of activated carbon was recorded in ball milling (4.841).

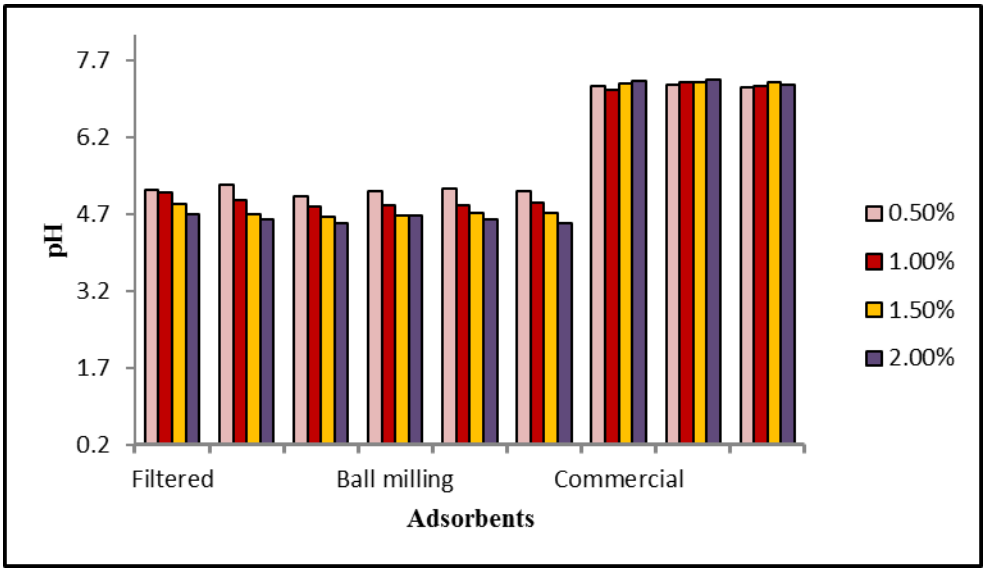
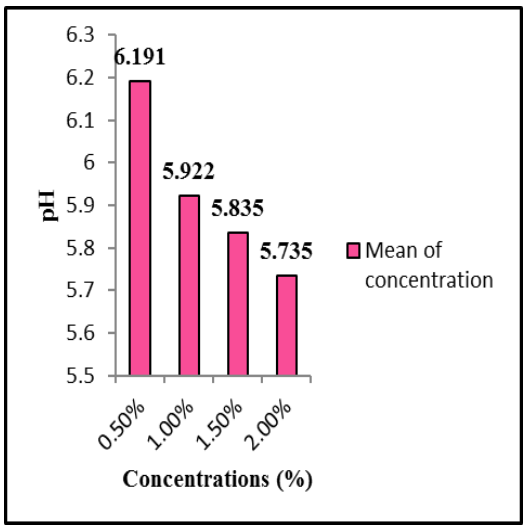


Figure 4.26 (a): pH of the effluent (2.5 N H_3PO_4 Mustard) activated carbon



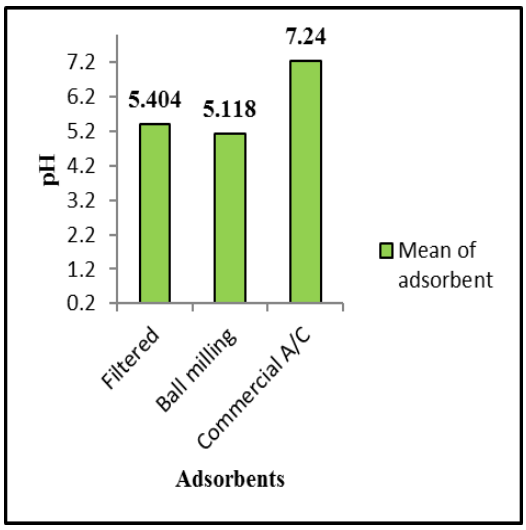


Figure 4.26 (b): Mean of concentration **Figure 4.26 (c): Mean of activated carbon**

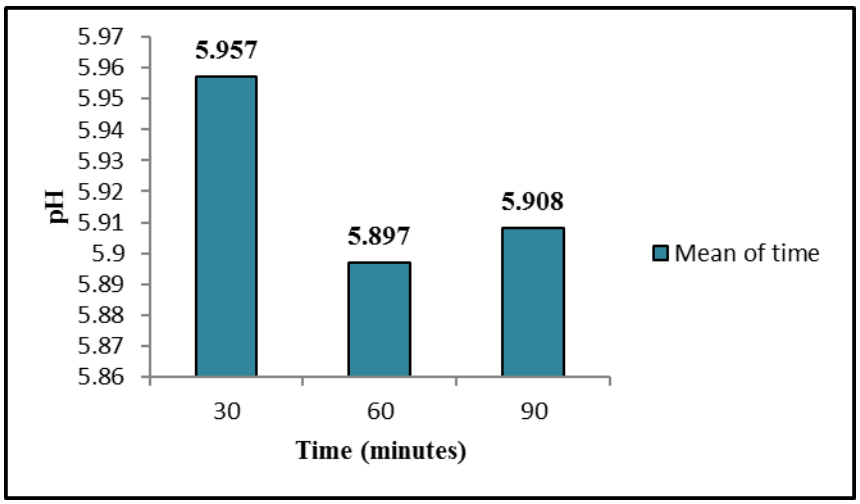


Figure 4.26 (d): Mean of time

Suresh (2015) worked on treatment of textile dye containing effluents found that there was higher biosorption capacity at lower pH, which might be due to electrostatic attraction between negatively charged dye anions and positively charged cell surface. Various researchers have reported that, decrease in pH enhanced the biosorption capacity of biomass for anionic dyes Iscen *et al.*, (2007), Won *et al.*, (2006), Nacera (2006), Aksu (2006) and Hu (1996). On the other hand, for cationic dyes, color removal decreased with a decrease in pH value due to repulsive forces between dye cations in solution and positively charged biosorbent surface low says Ncibi *et al.*, (2007), (1995) and Mittal and Gupta (1996).

4.12 pH of Metal complex dye effluent

4.12.1 pH of the effluent metal complex dye using 2.5 N H_2SO_4 Almond activated carbon

Table 4.19: pH of the effluent 2.5 N H_2SO_4 Almond activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.1%	5.240	5.173	5.130	5.143	5.197	5.147	5.253	5.177	5.153	**5.179**	30	**5.310**
0.25%	5.247	5.160	5.217	5.163	5.173	5.207	5.403	5.223	5.273	**5.230**	60	**5.234**
0.5%	5.323	5.253	5.297	5.170	5.243	5.140	5.403	5.227	5.317	**5.264**	90	**5.256**
1.0%	5.517	5.373	5.547	5.317	5.297	5.233	5.540	5.317	5.413	**5.395**		
Mean of Activated carbon	**5.290**			**5.203**			**5.308**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0025**	**0.0050**
Time	**0.0025**	**0.0050**
Concentration	**0.0029**	**0.0058**
Activated carbon*time	**0.0044**	**0.0087**
Activated carbon* concentration	**0.0050**	**0.0100**
Time*concentration	**0.0050**	**0.0100**
Activated carbon* time*concentration	**0.0087**	**0.0174**

Foot note:- Data given in the table are mean of three replication

Table 4.19 and figure 4.27 (a-d) depicted that the pH was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, pH was decreased at 1.0% activated carbon. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_2SO_4 almond activated carbon.

In 2.5 N H_2SO_4 almond (filtered) activated carbon, pH were recorded (5.373) at 1.0% concentration in 60 minute followed by(5.253) at 0.5% concentration in 60 minute while the pH (5.160) at 0.25% concentration in 60 minute and (5.130) at 0.1% concentration in 90 minute of contact time respectively. In 2.5 N H_2SO_4 almond (ball milling) activated carbon, it was recorded (5.233) in 90 minute at 1.0% concentration, (5.140) in 90 minute at 0.5% concentration, (5.163) in 30 minute at 0.25% concentration and (5.143) in 30 minute of contact time period at 0.1% concentration. In the commercial activated carbon, pH were recorded (5.317) at 1.0% concentration in 60 minute, (5.227) at 0.5% concentration in 60 minute, (5.223) at 0.25% concentration in 60 minute and (5.153) at 0.1% concentration in 90 minute of contact time period respectively. In commercial activated carbon, the lowest value for the mean of concentration (0.1%) and the mean of activated carbon was recorded as 5.179 and 5.308 respectively. The lowest value for the mean of time was recorded as 5.234 at 60 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in filtered (5.203).

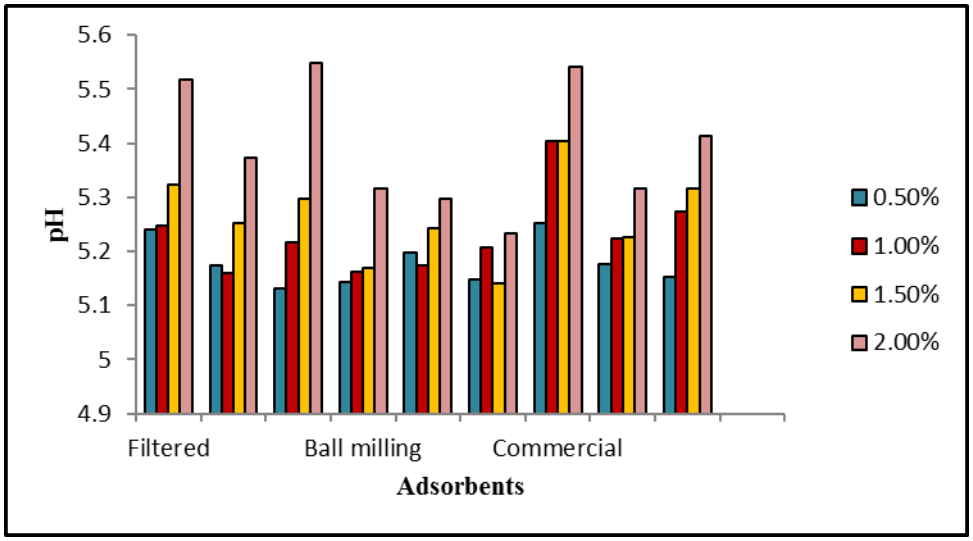


Figure 4.27 (a): pH of the effluent (2.5 N H_2SO_4 Almond) activated carbon

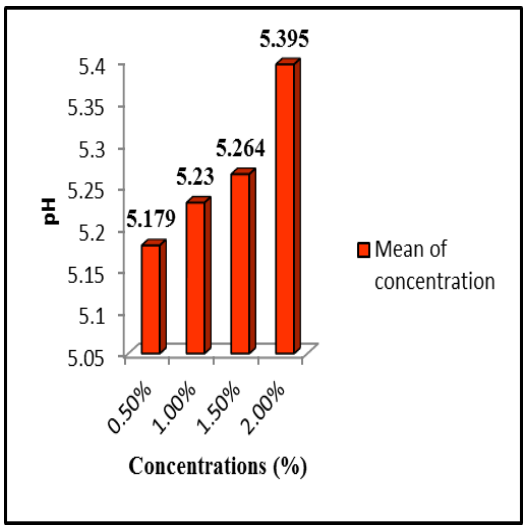


Figure 4.27 (b): Mean of concentration

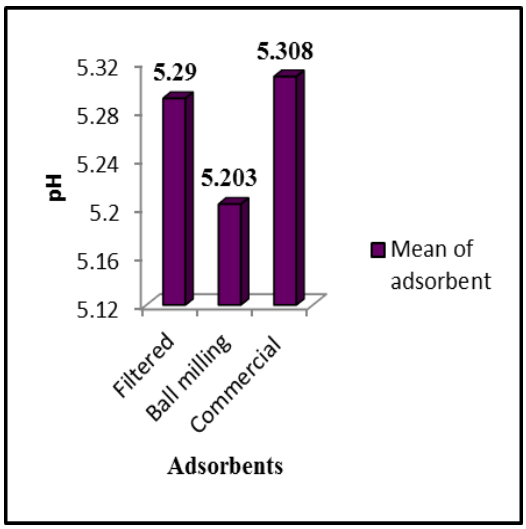


Figure 4.27 (c): Mean of activated carbon

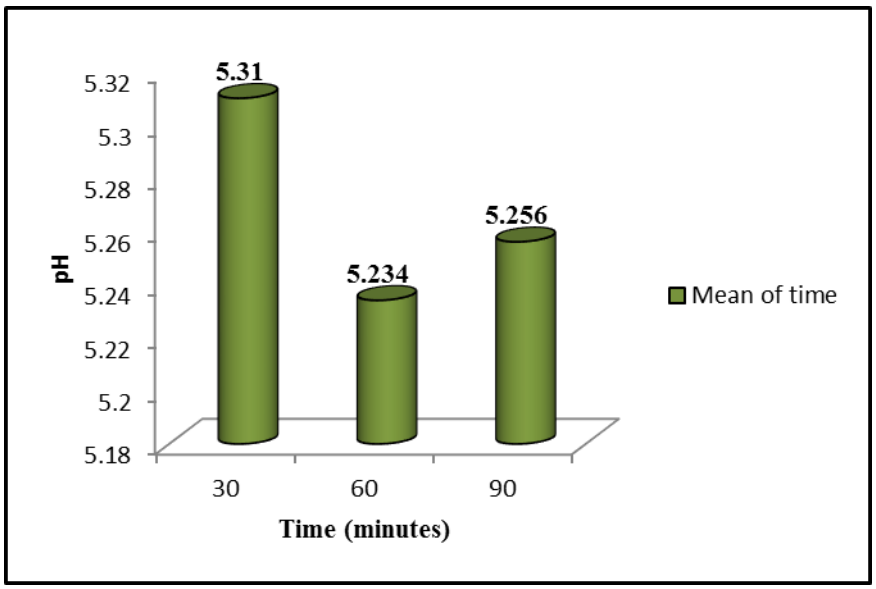


Figure 4.27 (d): Mean of time

Rao *et al.*, (2010) investigated on the effect of sodium dodecyl sulfate (SDS) on the adsorption of Zn (II) and Ni (II) on carbon derived from mustard oil cake (CMOC). They have found that at a lower pH value, the number of negatively charged surface sites decreases, which does not favor the adsorption of positively charged Zn (II) and Ni (II) ions. Lower adsorption of these metal ions in acidic pH is due to the presence of excess H+ ions competing with metal ions. The increased adsorption in the pH ranges from 4–6 because a solution of pH influences the adsorbed surface.

4.12.2 pH of the effluent metal complex dye using 2.5 N H_3PO_4 Rice Bran activated carbon

Table 4.20: pH of the effluent 2.5 N H_3PO_4 Rice Bran activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.1%	5.173	5.073	5.217	5.233	5.067	5.017	5.253	5.177	5.153	**5.151**	30	**5.179**
0.25%	5.193	5.153	5.147	5.063	5.040	5.017	5.403	5.223	5.273	**5.168**	60	**5.089**
0.5%	5.047	5.027	5.273	5.047	4.933	4.947	5.403	5.227	5.317	**5.136**	90	**5.135**
1.0%	4.917	4.963	5.077	4.880	4.867	4.773	5.540	5.317	5.413	**5.083**		
Mean of Activated carbon	**5.105**			**4.990**			**5.308**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0021**	**0.0043**
Time	**0.0021**	**0.0043**
Concentration	**0.0025**	**0.0049**
Activated carbon*time	**0.0037**	**0.0074**
Activated carbon* concentration	**0.0043**	**0.0086**
Time*concentration	**0.0043**	**0.0086**
Activated carbon* time*concentration	**0.0074**	**0.0148**

Foot note:- Data given in the table are mean of three replication

Table 4.20 and figure 4.28 (a-d) depicted that the pH was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, pH was decreased at 1.0% activated carbon. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 rice bran activated carbon.

The lowest pH recorded in 2.5 N H_3PO_4 rice bran (filtered) activated carbon were (4.917) in 1.0% concentration at 30 minute, (5.027) at 0.5% concentration in 60 minute, (5.147) at 0.25% concentration in 90 minute and (5.073) at 0.1% concentration in 60 minute of contact time period respectively. In case of 2.5 N H_3PO_4 rice bran (ball milling) activated carbon, it was recorded (4.773) at 1.0% concentration in 90 minute, (4.933) at 0.5% concentration in 60 minute, (5.017) at 0.25% concentration in 90 minute and (5.017) at 0.1% concentration in 90 minute of contact time period respectively. In the commercial activated carbon, (5.317) at 1.0% concentration in 60 minute, (5.227) at 0.5% concentration in 60 minute, (5.223) at 0.25% concentration in 60 minute and (5.153) at 0.1% concentration in 90 minute of contact time period respectively were recorded. In commercial activated carbon, the lowest value for the mean of concentration (1.0%) and the mean of activated carbon was recorded as 5.083 and 5.308 respectively. The lowest value for the mean of time was recorded as 5.089 at 60 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in ball milling (4.990).

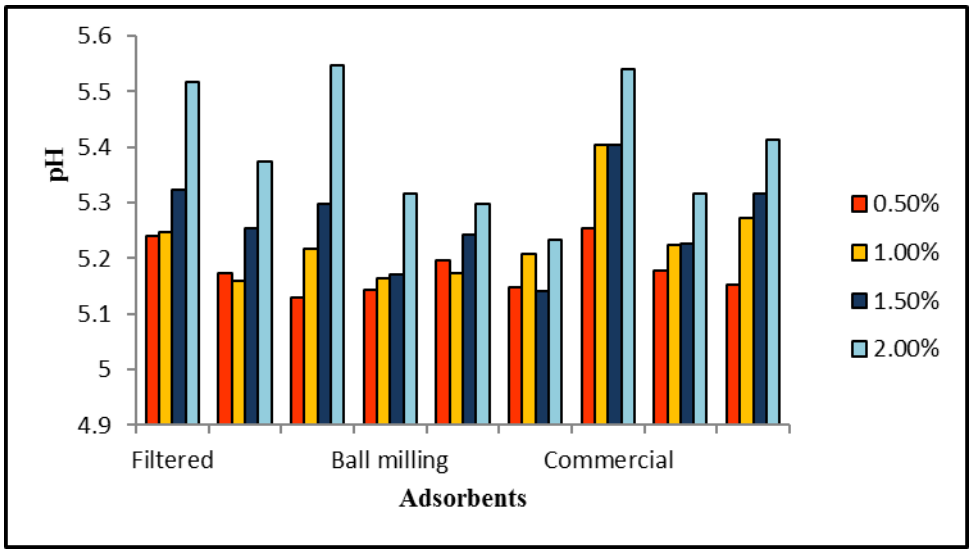


Figure 4.28 (a): pH the effluent (2.5 N H_3PO_4 Rice Bran) activated carbon

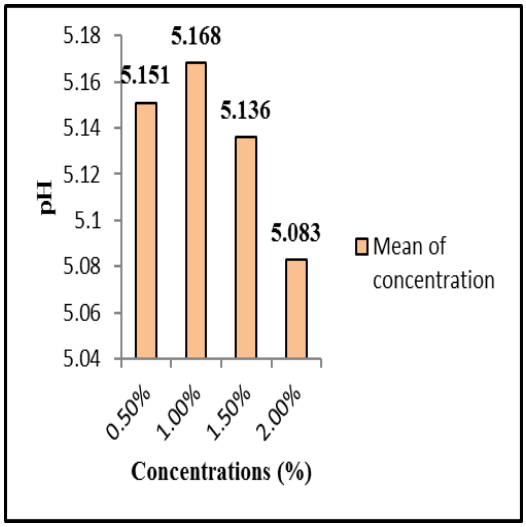


Figure 4.28 (b): Mean of concentration

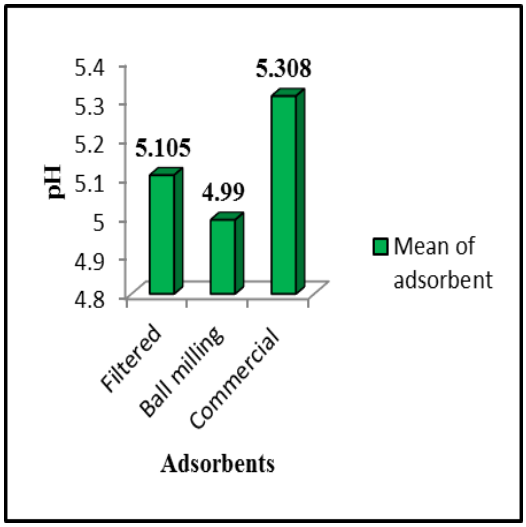


Figure 4.28 (c): Mean of activated carbon

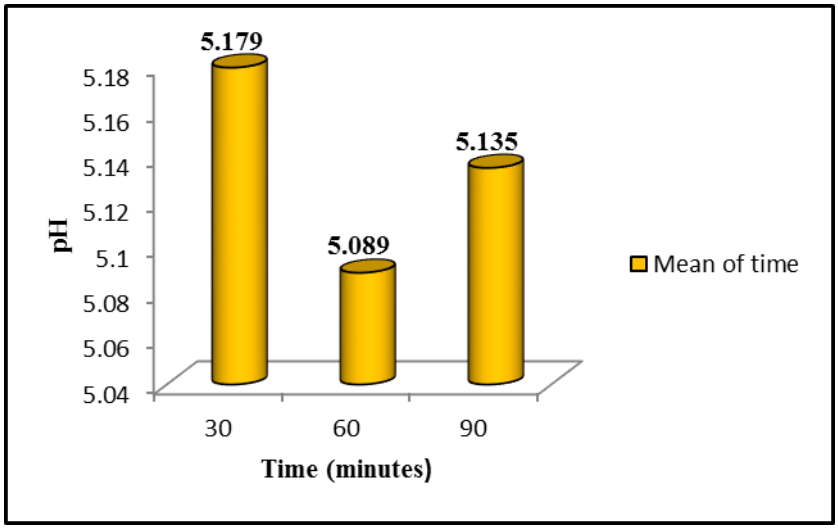


Figure 4.28 (d): Mean of time

Qodah and Shawabkah (2009) worked on production and characterization of granular activated carbon from activated sludge reported that the adsorption capacity increases significantly as pH decreases, which might be due to the fact that, at low pH the pesticide molecule get more charged sites which attracts the carbon surface leading to the adsorption of more pesticide molecules on the surface of the carbon.

4.12.3 pH of the effluent of metal complex dye using 2.5 N H_2SO_4 Sesame activated carbon

Table 4.21: pH of the effluent 2.5 N H_2SO_4 Sesame activated carbon

Activated carbon												
Concentration	**Filtered**			**Ball milling**			**Commercial**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.1%	5.237	5.197	5.417	5.073	5.287	5.267	5.253	5.177	5.153	**5.229**	30	**5.346**
0.25%	5.373	5.217	5.227	5.273	5.193	5.213	5.403	5.223	5.273	**5.266**	60	**5.239**
0.5%	5.287	5.357	5.337	5.473	5.140	5.227	5.403	5.227	5.317	**5.307**	90	**5.293**
1.0%	5.517	5.377	5.437	5.320	5.153	5.230	5.540	5.317	5.413	**5.367**		
Mean of Activated carbon	**5.331**			**5.238**			**5.308**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.0034**	**0.0067**
Time	**0.0034**	**0.0067**
Concentration	**0.0039**	**0.0078**
Activated carbon*time	**0.0058**	**0.0117**
Activated carbon* concentration	**0.0067**	**0.0135**
Time*concentration	**0.0067**	**0.0135**
Activated carbon* time*concentration	**0.0117**	**0.0233**

Foot note:- Data given in the table are mean of three replication

Table 4.21and figure 4.29 (a-d) showed that the pH was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, pH was decreased at 1.0% activated carbon. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_2SO_4 sesame activated carbon.

The lowest pH recorded were in 2.5 N H_2SO_4 sesame (filtered) activated carbon (5.377) at 1.0% concentration in 60 minute, (55.287) at 0.5% concentration in 30 minute, (5.217) at 0.25% concentration in 60 minute and (5.197) at 0.1% concentration in 60 minute of contact time period respectively. In 2.5 N H_2SO_4 sesame (ball milling) activated carbon, it was recorded (5.153) at 1.0% concentration in 60 minute, (5.140) at 0.5% concentration in 60 minute, (5.193) at 0.25% concentration in 60 minute and (5.073) at 0.1% concentration in 30 minute of contact time period respectively. In case of commercial activated carbon, pH were recorded as (5.317) at 1.0% concentration in 60 minute, (5.227) at 0.5% concentration in 60 minute, (5.223) at 0.25% concentration in 60 minute and (5.153) at 0.1% concentration in 90 minute of contact time period respectively. In commercial activated carbon, the lowest value for the mean of concentration (0.1%) and the mean of activated carbon was recorded as 5.229 and 5.308 respectively. The lowest value for the mean of time was recorded as 5.239 at 60 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in ball milling (5.238).

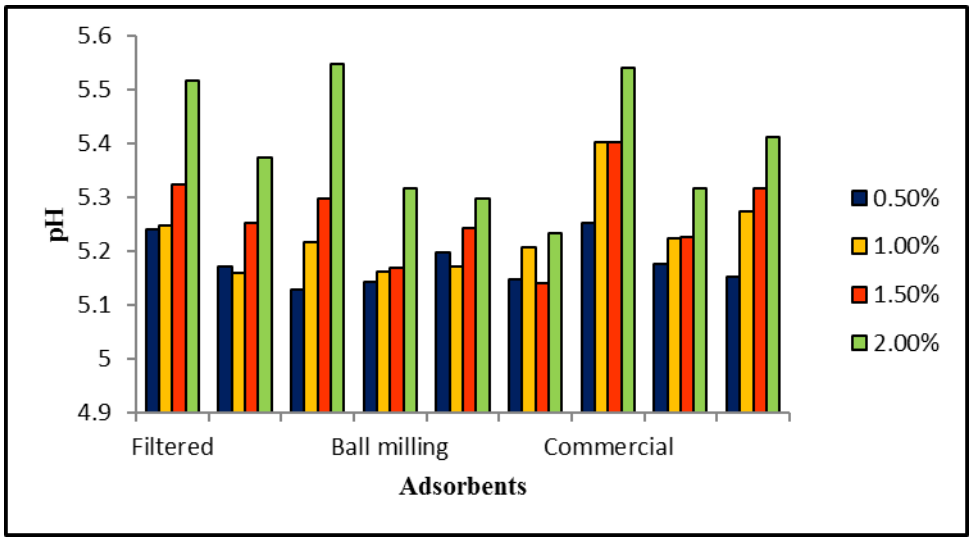


Figure 4.29 (a): pH of the effluent (2.5 N H_2SO_4 Sesame) activated carbon

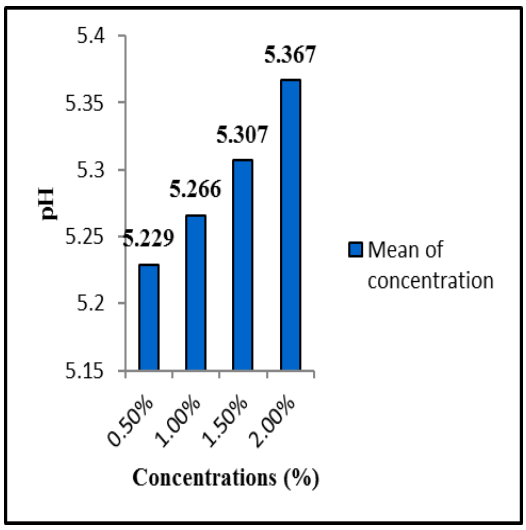


Figure 4.29 (b): Mean of concentration

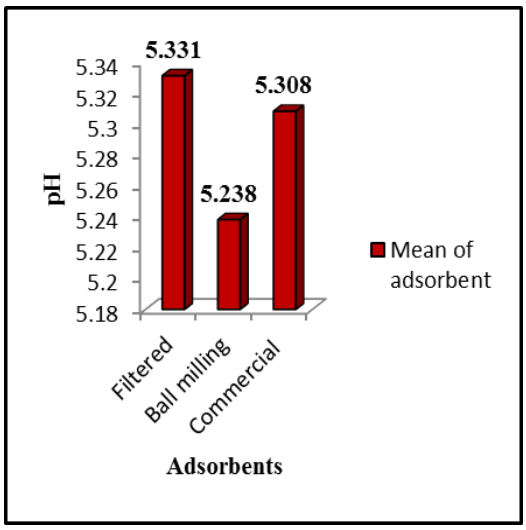


Figure 4.29 (c): Mean of activated carbon

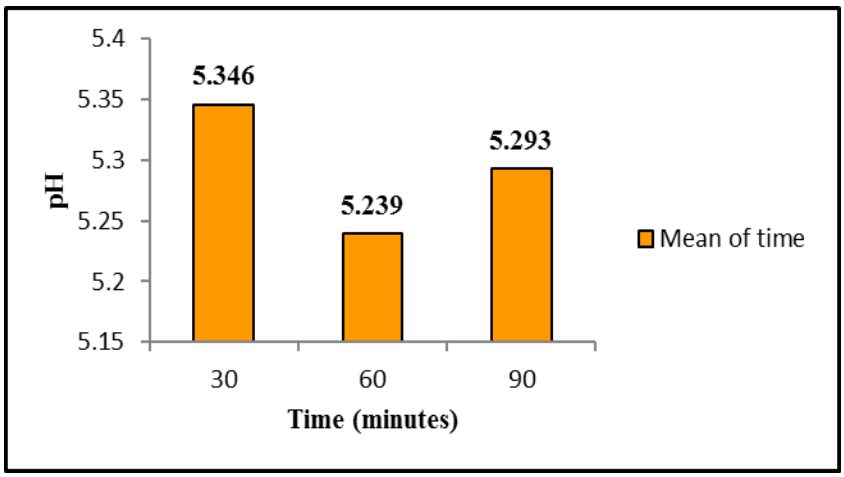


Figure 4.29 (d): Mean of time

Hameed (2013) had studied on the potential use of activated carbons such as amla seed carbon (ASC), jambul (jamun) seed carbon (JSC), tamarind seed carbon (TSC) and soap nut carbon (SNC) for the removal of chromotrope (CH) dye from simulated wastewater found that, highest percentage removal of CH (74%) was observed for ASC at pH 2 among all the low cost carbons. Similar type of report was given by Kavitha and Namsivayam (2008) for the adsorption of acid brilliant blue dye. It might be due to the fact that at a low pH value, the surfaces of the activated carbon become positively charged that results in strong electrostatic attraction between the positively charged carbon surface and anionic dye molecule leading to maximum adsorption of chromotrope.

4.13 TDS of acid dye treated sample

4.13.1 TDS of acid dye treated sample using 2.5 N H_3PO_4 Almond activated carbon

Table 4.22: TDS of the effluent 2.5 N H_3PO_4 Almond activated carbon

Activated carbon												
Concentration	**Filtered (mg/l)**			**Ball milling (mg/l)**			**Commercial (mg/l)**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.5%	2696.00	2699.33	2653.33	2739.33	2809.00	2689.00	2769.33	2799.66	2817.33	**2741.37**	30	**2693.27**
1.0%	2637.00	2825.66	2676.66	2546.66	2759.66	2649.33	2789.33	2779.33	2800.00	**2718.18**	60	**2744.94**
1.5%	2658.00	2695.33	2677.33	2599.6	2659.33	2659.33	2818.66	2819.33	2819.33	**2711.81**	90	**2726.19**
2.0%	2659.66	2626.66	2838.66	2606.33	2668.66	2664.66	2799.33	2797.33	2769.33	**2714.51**		
Mean of Activated carbon	**2695.30**			**2670.91**			**2798.19**					

	S. Ed(±)	**CD (0.05)**
Activated carbon	**0.6643**	**1.3250**
Time	**0.6643**	**1.3250**
Concentration	**0.7671**	**1.5300**
Activated carbon*time	**1.1507**	**2.2950**
Activated carbon* concentration	**1.3287**	**2.6500**
Time*concentration	**1.3287**	**2.6500**
Activated carbon* time*concentration	**2.3014**	**4.5899**

Foot note:- Data given in the table are mean of three replication

Table 4.22 and figure 4.30 (a-d) showed that the TDS was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, TDS was decreased at 2.0% activated carbon. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 almond activated carbon.

The lowest TDS were recorded in 2.5 N H_3PO_4 almond (filtered) activated carbon (2659.66mg/l) at 2.0% concentration in 30 minute followed by (2658.00mg/l) at 1.5% concentration in 30 minute, (2637.00mg/l) at 1.0% concentration in 30 minute and (2653.33mg/l) at 0.5% concentration in 90 minute of contact time period respectively. In 2.5 N H_3PO_4 almond (ball milling) activated carbon, it was recorded as (2606.33mg/l) at 2.0% concentration in 30 minute followed by (2599.6mg/l) at 1.5% concentration in 30 minute, (2546.66mg/l) at 1.0% concentration in 30 minute and (2689.00mg/l) at 0.5% concentration in 90 minute of contact time period respectively. In the commercial activated carbon, the TDS were recorded as (2769.33mg/l) at 2.0% concentration in 90 minute, (2818.66mg/l) at 1.5% concentration in 30 minute, (2779.33mg/l) at 1.0% concentration in 60 minute and (2769.33mg/l) at 0.5% concentration in 30 minute of contact time period respectively. The lowest value for the TDS mean of concentration (1.5%) and the mean of activated carbon was recorded as (2711.81mg/l) and (2798.19mg/l) respectively in commercial activated carbon. The lowest value for the TDS mean of time was recorded as (2693.27mg/l) at 30 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the TDS mean of activated carbon was recorded in ball milling (2670.9 mg/l).

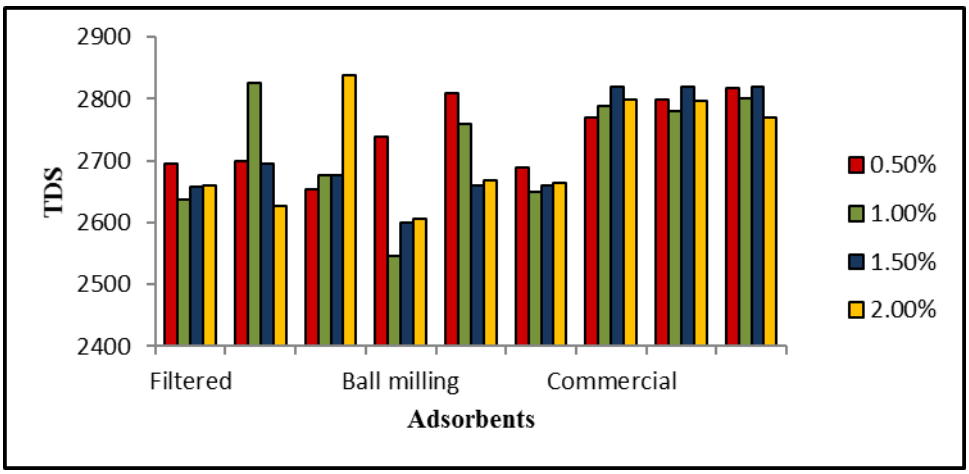
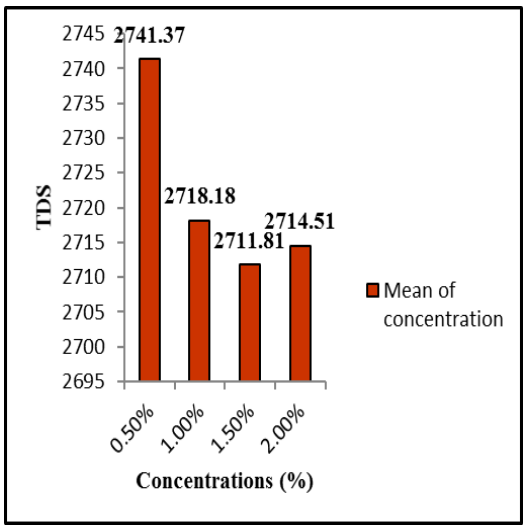


Figure 4.30 (a): TDS of the effluent (2.5 N H_3PO_4 Almond) activated carbon



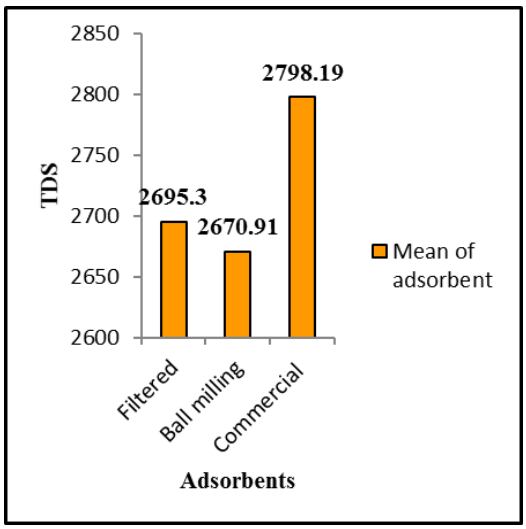


Figure 4.30 (b): Mean of concentration **Figure 4.30 (c): Mean of activated carbon**

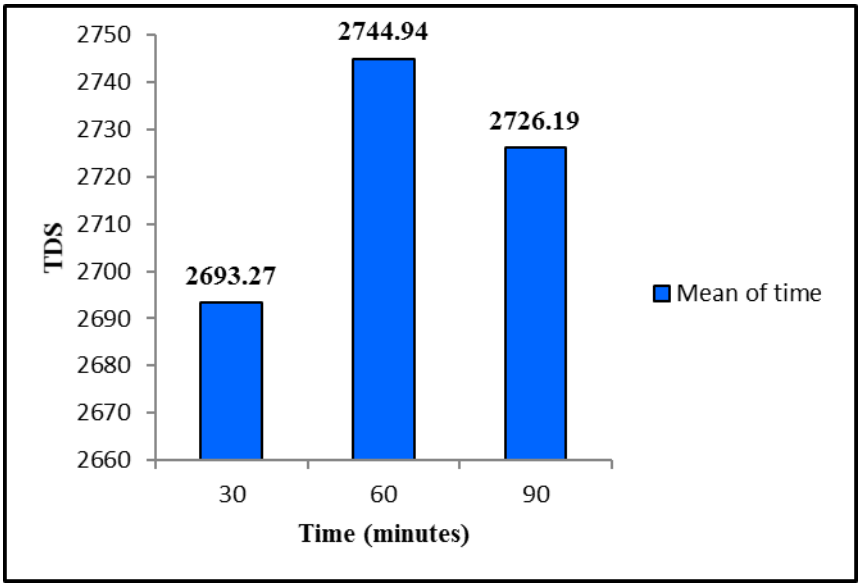


Figure 4.30 (d): Mean of time

Elango and Govindasamy (2018) have studied the characteristics of Total Dissolved Solids (TDS) of activated carbon from temple waste flowers prepared by direct pyrolysis process. They have found that with sodium sulphate (Na_2SO_4) and potassium hydroxide (KOH) treatment, TDS was found to be 18220 mg/L which was much higher due to the fixing, bleaching and dyeing agents and it may cause a salinity problem.

4.13.2 TDS of treated sample acid dye using 1 N H_3PO_4 Coconut activated carbon

Table 4.23: TDS of the effluent 1 N H_3PO_4 Coconut activated carbon

Activated carbon												
Concentration	**Filtered (mg/l)**			**Ball milling (mg/l)**			**Commercial (mg/l)**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.5%	2429.33	2488.00	2538.66	2591.33	2628.66	2618.66	2769.33	2799.66	2817.33	**2631.22**	30	**2634.77**
1.0%	2409.33	2479.33	2509.66	2544.66	2539.33	2578.66	2789.33	2779.33	2800.00	**2603.29**	60	**2686.25**
1.5%	2478.66	2799.33	2548.33	2729.33	2871.33	2599.33	2818.66	2819.33	2819.33	**2720.40**	90	**2646.41**
2.0%	2589.33	2598.66	2549.00	2668.66	2634.66	2608.66	2799.33	2797.33	2769.33	**2668.33**		
Mean of Activated carbon	**2534.80**			**2634.44**			**2798.19**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**22.6156**	**45.1055**
Time	**22.6156**	**NS**
Concentration	**26.1143**	**52.0833**
Activated carbon*time	**39.1715**	**NS**
Activated carbon* concentration	**45.2313**	**NS**
Time*concentration	**45.2313**	**90.2110**
Activated carbon* time*concentration	**78.3429**	**NS**

Foot note:- Data given in the table are mean of three replication

Table 4.23 and figure 4.31 (a-d) showed that the TDS was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, TDS was decreased at 2.0% activated carbon. From the statistical analysis, it revealed that all the factors and their interactions except time, activated carbon*time, activated carbon* concentration and activated carbon* time*concentration were found to be non significant at 5% level with respect to 1 N H_3PO_4 coconut activated carbon.

The lowest TDS recorded in 1 N H_3PO_4 coconut (filtered) activated carbon were (2549.00mg/l) at 2.0% concentration in 90 minute, (2478.66mg/l) at 1.5% concentration in 30 minute, (2409.33mg/l) at 1.0% concentration in 30 minute and (2429.33mg/l) at 0.5% concentration in 30 minute of contact time period respectively. In 1 N H_3PO_4 coconut (ball milling) activated carbon, TDS were recorded as (2608.66mg/l) at 2.0% concentration in 90 minute, (2599.33mg/l) at 1.5% concentration in 90 minute, (2539.33mg/l) at 1.0% concentration in 60 minute and (2591.33mg/l) at 0.5% concentration in 30 minute of contact time period respectively. In the commercial activated carbon, TDS were recorded as (2769.33mg/l) at 2.0% concentration in 90 minute, (2818.66mg/l) at 1.5% concentration in 30 minute, (2779.33mg/l) at 1.0% concentration in 60 minute and (2769.33mg/l) at 0.5% concentration in 30 minute of contact time period respectively. The lowest value for the TDS mean of concentration at 1.0% and the mean of activated carbon was recorded as (2603.29mg/l) and (2798.19mg/l) respectively in commercial activated carbon. The lowest value for the TDS mean of time was recorded as (2634.77mg/l) at 30 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the TDS mean of activated carbon was recorded in filtered activated carbon (2534.80mg/l).

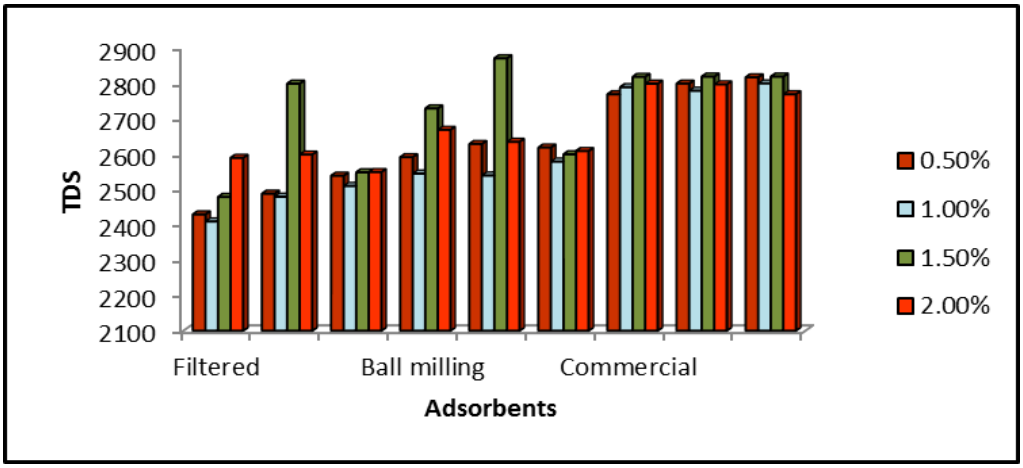
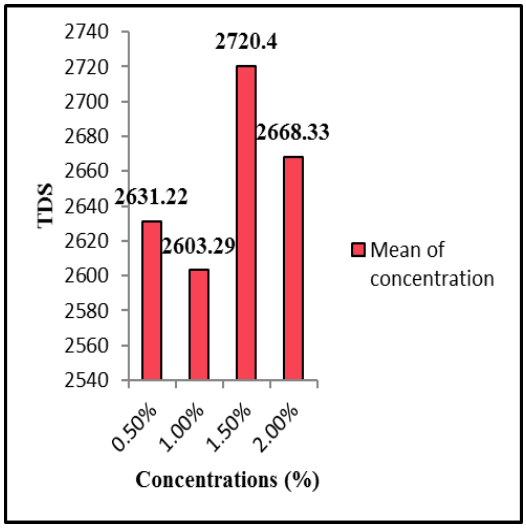


Figure 4.31 (a): TDS of the effluent (1 N H_3PO_4 Coconut) activated carbon



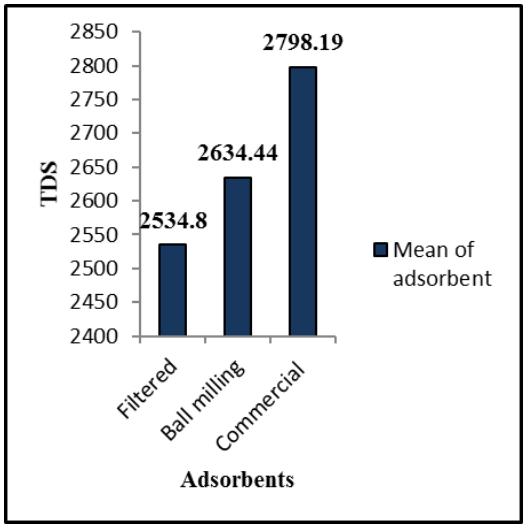


Figure 4.31 (b): Mean of concentration

Figure 4.31(c): Mean of activated carbon

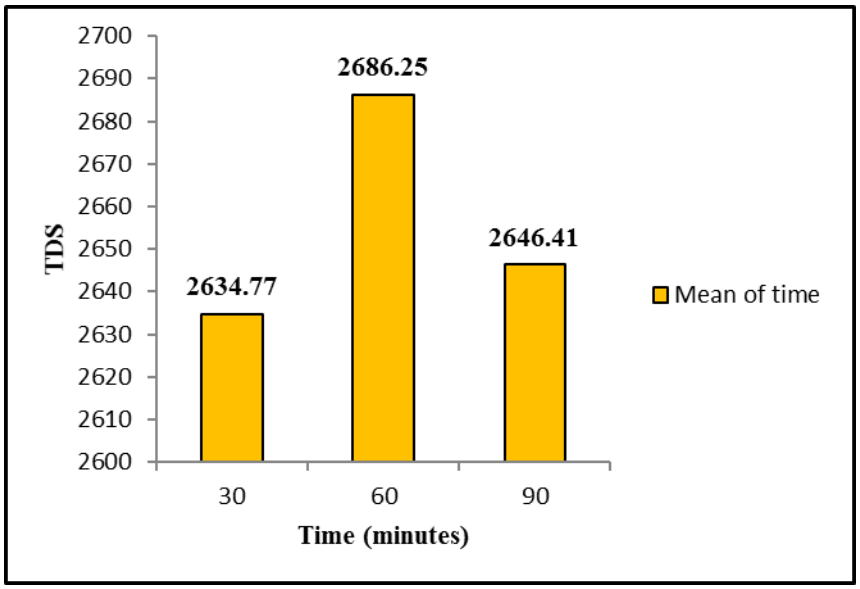


Figure 4.31 (d): Mean of time

4.13.3 TDS of treated sample acid dye using 2.5 N H_3PO_4 Mustard activated carbon

Table 4.24: TDS of the effluent 2.5 N H_3PO_4 Mustard activated carbon

Activated carbon												
Concentration	**Filtered (mg/l)**			**Ball milling (mg/l)**			**Commercial (mg/l)**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.5%	2878.66	2529.33	2589.33	2638.66	2638.66	2639.33	2769.33	2799.66	2817.33	**2700.03**	30	**2660.05**
1.0%	2469.33	2498.66	2479.33	2568.66	2628.66	2640.00	2789.33	2779.33	2800.00	**2628.14**	60	**2642.69**
1.5%	2466.66	2478.66	2519.33	2628.00	2618.00	2566.66	2818.66	2819.33	2819.33	**2637.18**	90	**2650.52**
2.0%	2464.66	2487.33	2518.66	2629.33	2637.33	2647.66	2799.33	2797.33	2769.33	**2639.00**		
Mean of Activated carbon	**2531.66**			**2623.41**			**2798.19**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.4456**	**0.8887**
Time	**0.4456**	**0.8887**
Concentration	**0.5145**	**1.0262**
Activated carbon*time	**0.7718**	**1.5393**
Activated carbon* concentration	**0.8912**	**1.7774**
Time*concentration	**0.8912**	**1.7774**
Activated carbon* time*concentration	**1.5436**	**3.0786**

Foot note:- Data given in the table are mean of three replication

Table 4.24 and figure 4.32 (a-d) showed that the TDS was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, TDS was decreased at 2.0% activated carbon. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 mustard activated carbon.

The lowest TDS was recorded in 2.5 N H_3PO_4 mustard (filtered) activated carbon, TDS were recorded as (2464.66mg/l) at 2.0% concentration in 30 minute followed by (2466.66mg/l) at 1.5% concentration in 30 minute, (2469.33mg/l) at 1.0% concentration in 30 minute and (2529.33mg/l) at 0.5% concentration in 60 minute of contact time period respectively. In 2.5 N H_3PO_4 mustard (ball milling) activated carbon, TDS were recorded as (2629.33mg/l) at 2.0% concentration in 30 minute followed by (2618.00mg/l) at 1.5% concentration in 60 minute, (2568.66mg/l) at 1.0% concentration in 30 minute and (2638.66mg/l) at 0.5% concentration in 30 minute and 60 minute of contact time period respectively. In the commercial activated carbon, TDS were recorded as (2769.33mg/l) at 2.0% concentration in 90 minute followed by (2818.66mg/l) at 1.5% concentration in 30 minute, (2779.33mg/l) at 1.0% concentration in 60 minute and (2769.33mg/l) at 0.5% concentration in 30 minute of contact time period respectively. The lowest value for the TDS mean of concentration at 1.0% and the mean of activated carbon was recorded as (2628.14mg/l) and (2798.19mg/l) respectively in commercial activated carbon. The lowest value for the TDS mean of time was recorded as (2642.69mg/l) at 60 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the TDS mean of activated carbon was recorded in filtered activated carbon (2531.66mg/l).

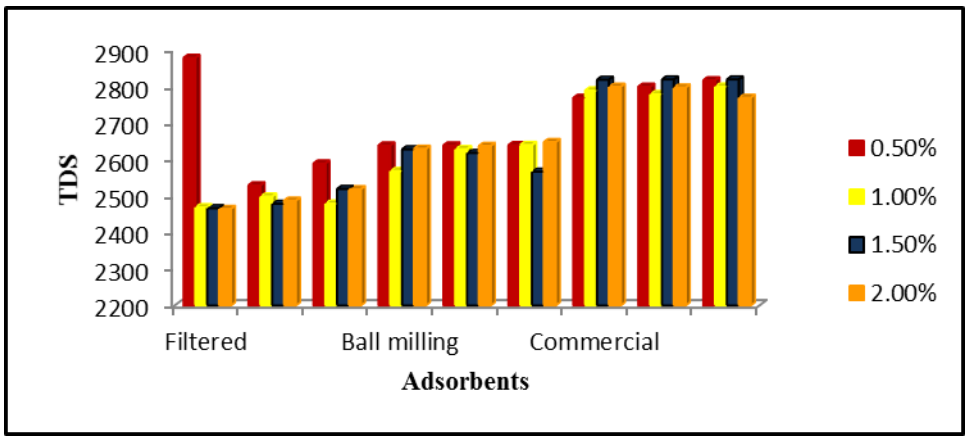


Figure 4.32 (a): TDS of the effluent (2.5 N H_3PO_4 Mustard) activated carbon

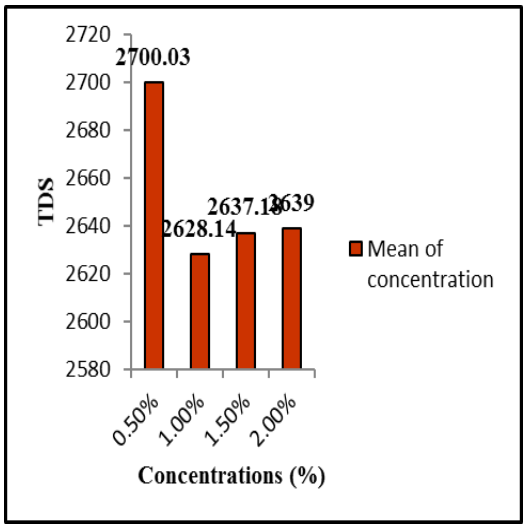


Figure 4.32 (b): Mean of concentration

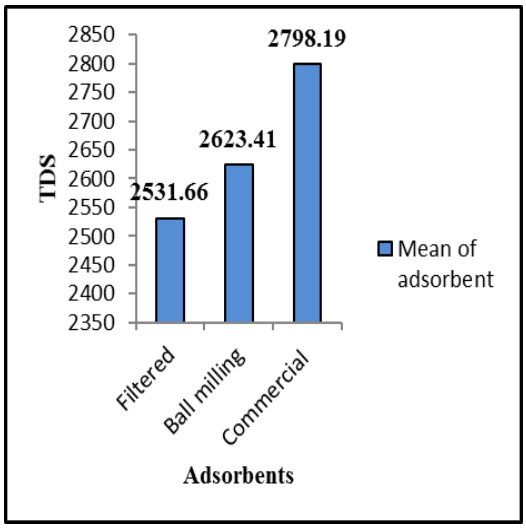


Figure 4.32 (c): Mean of activated carbon

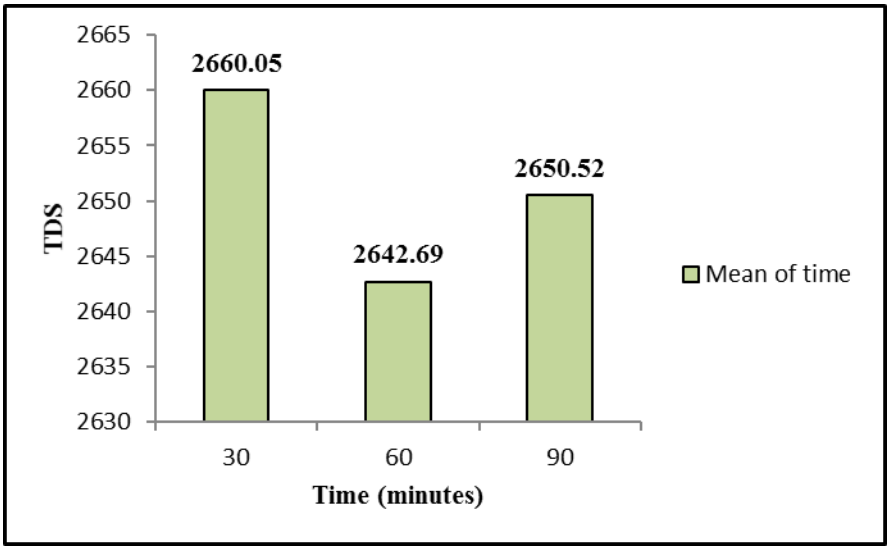


Figure 4.32 (d): Mean of time

Islam and Mostafa (2020) worked on characterization of the textile dyeing effluent to evaluate the color removal efficiency of the coagulant using polyaluminum chloride (PAC) found that that the TDS of the effluents was found to be higher indicated that the discharged effluents had the potential power to worsen the quality of surface water and aquatic life. Similar findings were made by Imtiazuddin *et al.*, (2012).

4.14 Removal of Metal complex dye effluent

4.14.1 TDS of treated sample metal complex dye using 2.5 N H_2SO_4 Almond activated carbon

Table 4.25: TDS of the effluent 2.5 N H_2SO_4 Almond activated carbon

Activated carbon												
Concentration	**Filtered (mg/l)**			**Ball milling (mg/l)**			**Commercial (mg/l)**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.1%	1709.33	1928.66	2046.66	1819.33	1928.00	1549.33	1989.66	1968.66	2049.00	**1887.63**	30	**2020.97**
0.25%	1849.33	1927.33	2089.33	1989.66	1889.33	1889.33	2049.00	1959.33	2059.66	**1966.92**	60	**1953.36**
0.5%	2149.33	2053.33	2049.33	2158.66	2129.33	1138.66	2059.66	1989.00	2069.00	**1977.37**	90	**1939.86**
1.0%	2148.66	2078.66	2128.66	2219.33	2139.33	2110.00	2109.66	1449.33	2099.33	**2053.66**		
Mean of Activated carbon	**2013.22**			**1913.36**			**1987.61**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**42.1999**	**NS**
Time	**42.1999**	**NS**
Concentration	**48.7282**	**97.1854**
Activated carbon*time	**73.0923**	**145.7781**
Activated carbon* concentration	**84.3998**	**168.3300**
Time*concentration	**84.3998**	**168.3300**
Activated carbon* time*concentration	**146.1847**	**291.5562**

Foot note:- Data given in the table are mean of three replication

Table 4.25 and figure 4.33 (a-d) showed that the TDS was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, TDS was decreased at 1.0% activated carbon. From the statistical analysis, it revealed that all the factors and their interactions except activated carbon and time were found to be non- significant at 5% level with respect to 2.5 N H_2SO_4 almond activated carbon.

In 2.5 N H_2SO_4 almond (filtered) activated carbon, TDS was recorded (2078.66) at 1.0% concentration in 60 minute, (2049.33mg/l) at 0.5% concentration in 90 minute, (1849.33mg/l) at 0.25% concentration in 30 minute and (1709.33mg/l) at 0.1% concentration in 30 minute respectively. In 2.5 N H_2SO_4 almond (ball milling) activated carbon, TDS was recorded (2110.00mg/l) at 1.0% concentration in 90 minute, (1138.66mg/l) at 0.5% concentration in 90 minute, (1889.33mg/l) at 0.25% concentration in 60 minute and 90 minute and (1549.33mg/l) at 0.1% concentration in 90 minute of contact time period respectively. In the commercial activated carbon, TDS was recorded as (1449.33mg/l) at 1.0% concentration in 60 minute, (1989.00mg/l) at 0.5% concentration in 60 minute, (1959.33mg/l) at 0.25% concentration in 60 minute and (1968.66mg/l) at 0.1% concentration in 60 minute of contact time period respectively. In commercial activated carbon, the lowest value for the mean of concentration at 0.1% and the mean of activated carbon was recorded as (1887.63mg/l) and (1987.61mg/l) respectively. The lowest value for the mean of time was recorded as (1939.86mg/l) at 90 minutes. Between the activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in ball milling activated carbon0 (1913.36mg/l).

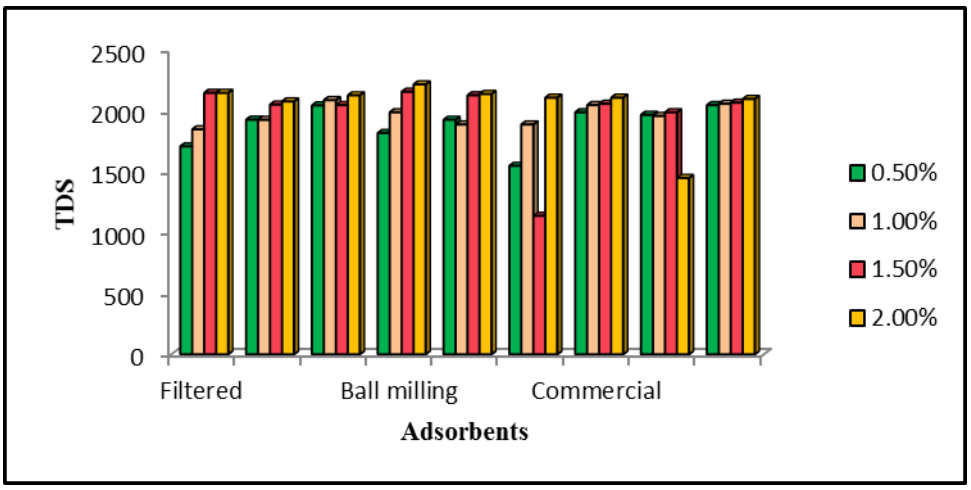


Figure 4.33 (a): TDS of the effluent (2.5 N H_2SO_4 Almond) activated carbon

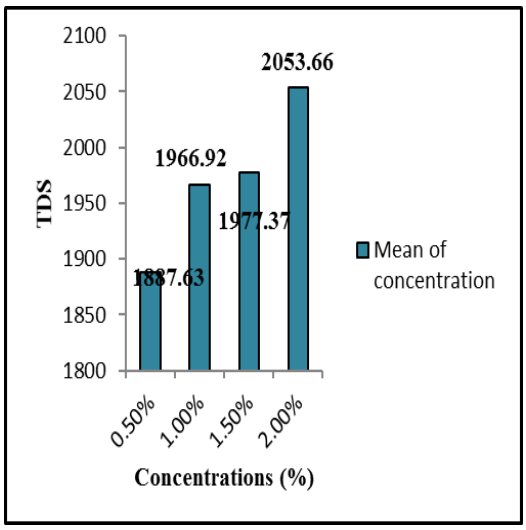


Figure 4.33 (b): Mean of concentration

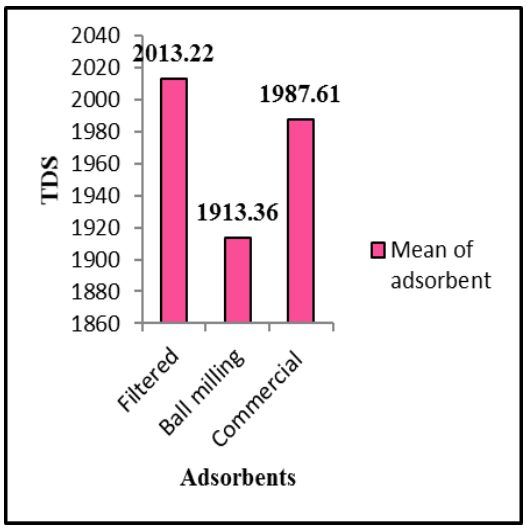


Figure 4.33 (c): Mean of activated carbon

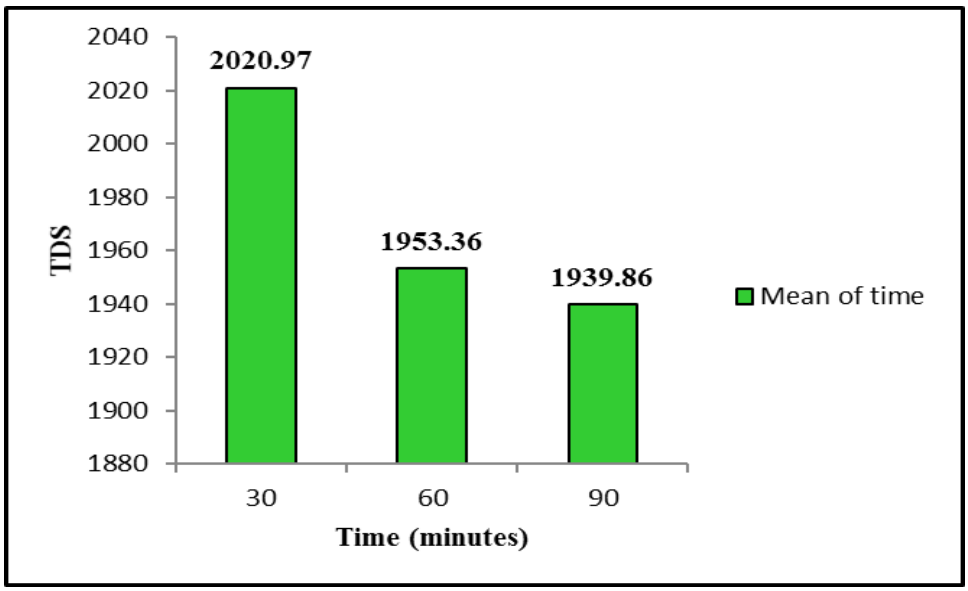


Figure 4.33 (d): Mean of time

Vasanthy *et al.,* (2008) studied on the removal and reduction of total dissolved solids (TDS) of the textile dyeing effluent using oxidizing agents, coagulating agents and adsorbent individually with different combinations found that 80% TDS was removed using 0.25g of oxidizing agent in one hour whereas 1 g of coagulating agent removed TDS 90%. With the combination of sodium hypochlorite, commercial activated carbon (CAC) and alum, the removal of TDS was found to be 79%.

4.14.2 TDS of treated sample metal complex dye using 2.5 N H_3PO_4 Rice Bran activated carbon

Table 4.26: TDS of the effluent 2.5 N H_3PO_4 Rice Bran activated carbon

Activated carbon												
Concentration	**Filtered (mg/l)**			**Ball milling (mg/l)**			**Commercial (mg/l)**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.1%	2049.33	1889.33	2068.66	2049.66	2049.33	2047.33	1989.66	1968.66	2049.00	**2017.88**	30	**1996.08**
0.25%	1988.66	2049.33	2060.00	1959.00	1989.33	1989.33	2049.00	1959.33	2059.66	**2011.51**	60	**1938.38**
0.5%	1889.33	1989.00	2159.33	1960.00	1939.33	1989.33	2059.66	1989.00	2069.00	**2004.88**	90	**2028.30**
1.0%	1870.00	2048.66	1859.33	1979.00	1940.00	1889.33	2109.66	1449.33	2099.33	**1916.07**		
Mean of Activated carbon	**1993.41**			**1981.75**			**1987.61**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**42.7498**	**NS**
Time	**42.7498**	**NS**
Concentration	**49.3633**	**NS**
Activated carbon*time	**74.0449**	**NS**
Activated carbon* concentration	**85.4997**	**NS**
Time*concentration	**85.4997**	**NS**
Activated carbon* time*concentration	**148.0898**	**NS**

Foot note:- Data given in the table are mean of three replication

Table 4.26 and figure 4.34 (a-d) showed that the TDS was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, TDS was decreased at 1.0% activated carbon. From the statistical analysis, it revealed that all the factors and their interactions were found to be non- significant at 5% level with respect to 2.5 N H_3PO_4 rice bran activated carbon.

In 2.5 N H_3PO_4 rice bran (filtered) activated carbon, TDS was recorded (1859.33) at 1.0% concentration in 90 minute, (1889.33mg/l) at 0.5% concentration in 30 minute, (1988.66mg/l) at 0.25% concentration in 30 minute and (1889.33mg/l) at 0.1% concentration in 60 minute of contact time period respectively. In 2.5 N H_3PO_4 rice bran (ball milling) activated carbon, TDS was recorded (1889.33mg/l) at 1.0% concentration in 90 minute, (1939.33mg/l) at 0.5% concentration in 60 minute, (1959.00mg/l) at 0.25% concentration in 30 minute and (2047.33mg/l) at 0.1% concentration in 90 minute of contact time period. In the commercial activated carbon, TDS was recorded as (1449.33mg/l) at 1.0% concentration in 60 minute, (1989.00mg/l) at 0.5% concentration in 60 minute, (1959.33mg/l) at 0.25% concentration in 60 minute and (1968.66mg/l) at 0.1% concentration in 60 minute of contact time period respectively. In commercial activated carbon, the lowest value for the mean of concentration (1.0%) and the mean of activated carbon was recorded as (1916.07mg/l) and (1987.61mg/l) respectively. The lowest value for the mean of time was recorded as (1938.38mg/l) at 60 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in ball milling activated carbon (1981.75mg/l).

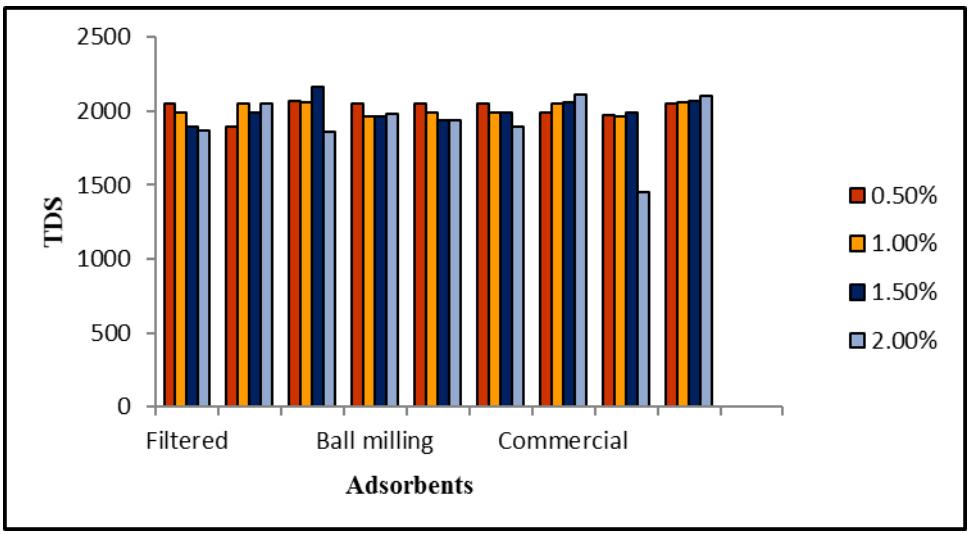


Figure 4.34 (a): TDS of the effluent (2.5 N H_3PO_4 Rice Bran) activated carbon

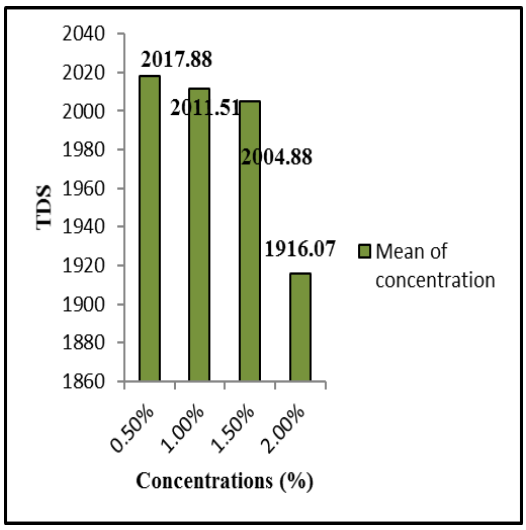


Figure 4.34 (b): Mean of concentration

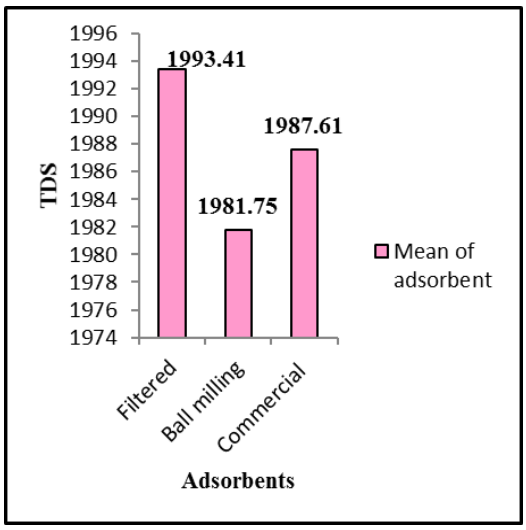


Figure 4.34 (c): Mean of activated carbon

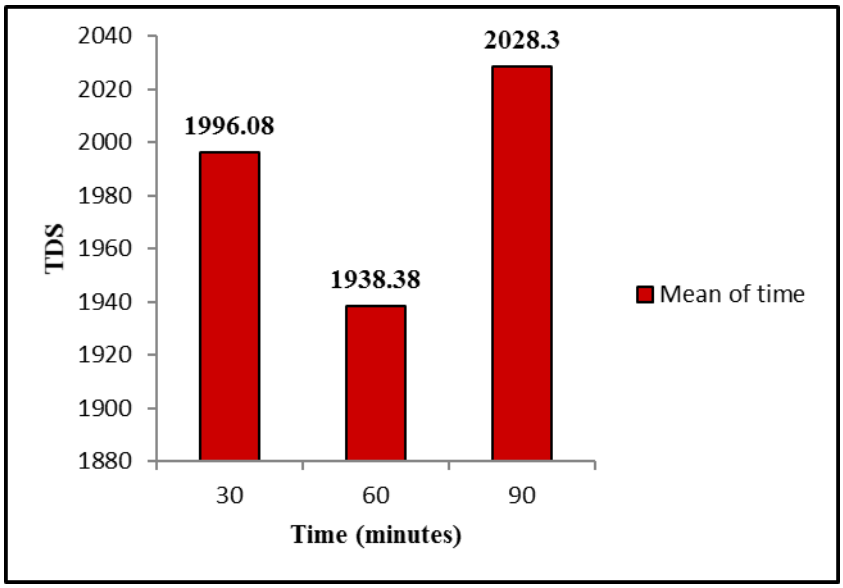


Figure 4.34 (d): Mean of time

4.14.3 TDS of treated sample metal complex dye using 2.5 N H_2SO_4 Sesame activated carbon

Table 4.27: TDS of the effluent 2.5 N H_2SO_4 Sesame activated carbon

Activated carbon												
Concentration	**Filtered(mg/l)**			**Ball milling(mg/l)**			**Commercial(mg/l)**			**Mean of concentration**	**Time in minutes**	**Mean of time**
	30 minute	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**	**30 minute**	**60 minute**	**90 minute**			
0.1%	2049.66	2079.66	2119.33	2099.66	2148.66	2099.33	1989.66	1968.66	2049.00	**2067.07**	30	**2174.41**
0.25%	2129.66	2179.66	2189.66	2179.33	2259.00	2179.66	2049.00	1959.33	2059.66	**2131.66**	60	**2164.75**
0.5%	2239.33	2289.33	2329.33	2309.33	2289.33	2330.00	2059.66	1989.00	2069.00	**2211.59**	90	**2194.13**
1.0%	2359.33	2418.66	2428.66	2518.66	2319.66	2376.66	2109.66	2076.00	2099.33	**2300.74**		
Mean of Activated carbon	**2234.36**			**2259.11**			**1987.61**					

	S. Ed(±)	**CD(0.05)**
Activated carbon	**0.4964**	**0.9900**
Time	**0.4964**	**0.9900**
Concentration	**0.5732**	**1.1432**
Activated carbon*time	**0.8598**	**1.7147**
Activated carbon* concentration	**0.9928**	**1.9800**
Time*concentration	**0.9928**	**1.9800**
Activated carbon* time*concentration	**1.7195**	**3.4295**

Foot note:- Data given in the table are mean of three replication

Table 4.27 and figure 4.35 (a-d) showed that the TDS was decreased with the increased of activated carbon. In addition to this, the table also revealed that among all the concentrations, TDS was decreased at 1.0% activated carbon. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_2SO_4 sesame activated carbon.

The lowest TDS were recorded in 2.5 N H_2SO_4 sesame (filtered) activated carbon (2359.33mg/l) at 1.0% concentration in 30 minute (2239.33mg/l) at 0.5% concentration in 30 minute (2129.66mg/l) at 0.25% concentration in 30 minute and (2049.66mg/l) at 0.1% concentration in 30 minute of contact time period respectively. In 2.5 N H_2SO_4 sesame (ball milling) activated carbon, TDS were recorded as (2319.66mg/l) at 1.0% concentration in 60 minute, (2309.33mg/l) at 0.5% concentration in 30 minute, (2179.33mg/l) at 0.25% concentration in 30 minute and (2099.33mg/l) at 0.1% concentration in 90 minute of contact time period. In the commercial activated carbon, TDS was recorded as (1449.33mg/l) at 1.0% concentration in 60 minute, (1989.00mg/l) at 0.5% concentration in 60 minute, (1959.33mg/l) at 0.25% concentration in 60 minute and (1968.66mg/l) at 0.1% concentration in 60 minute of contact time period respectively. In commercial activated carbon, the lowest value for the mean of concentration at 0.1% and the mean of activated carbon was recorded as (2067.07mg/l) and (1987.61mg/l) respectively. The lowest value for the mean of time was recorded as (2164.75mg/l) at 60 minutes. Between the two activated carbon (filtered and ball milling), the lowest value for the mean of activated carbon was recorded in filtered activated carbon (2234.36mg/l).

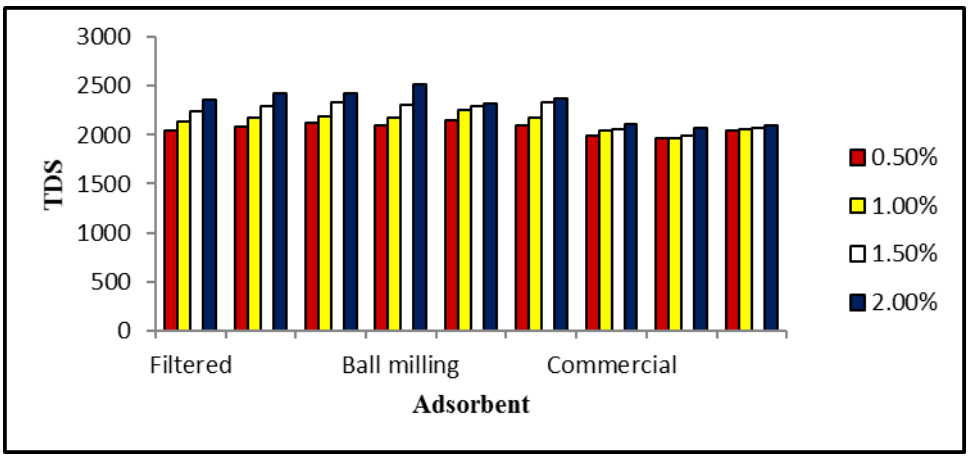


Figure 4.35 (a): TDS of the effluent (2.5 N H_2SO_4 Sesame) activated carbon

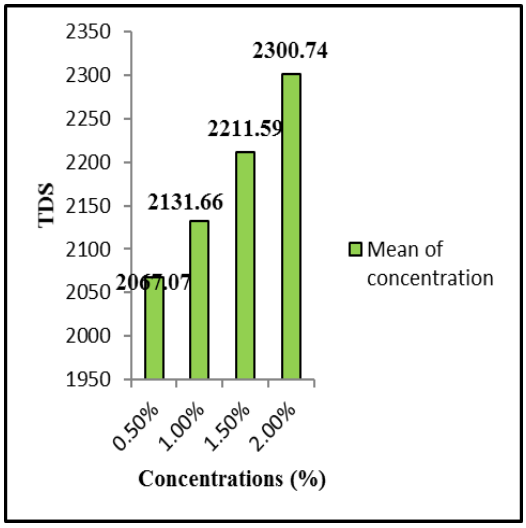


Figure 4.35 (b): Mean of concentration

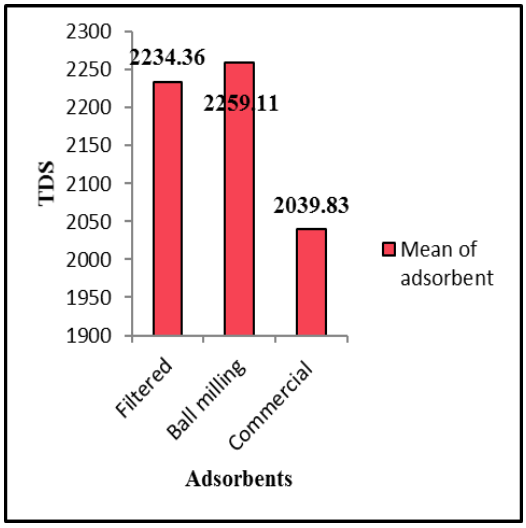


Figure 4.35 (c): Mean of activated carbon

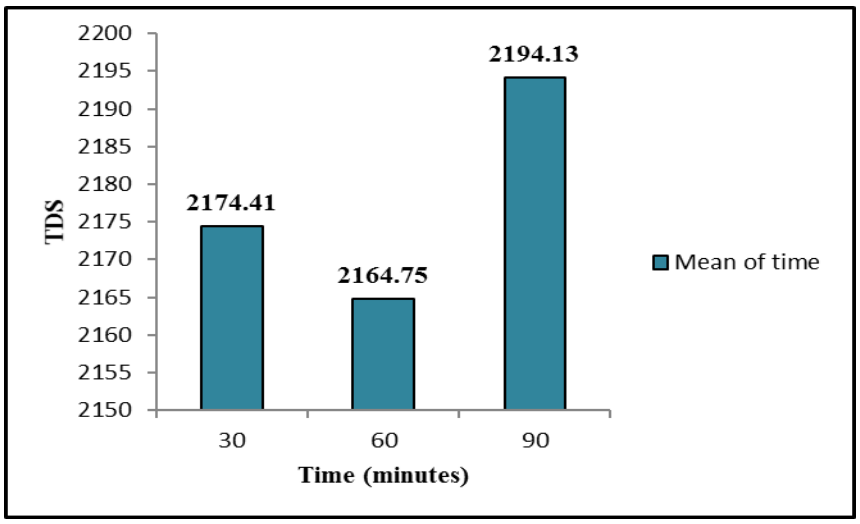


Figure 4.35 (d): Mean of time

Prasanthrajan (2019) studied on reduction of total dissolved solids (TDS) in textile effluent found that removal of TDS using biological and chemical method were not found to be successful as they were not effective in TDS reduction. The Physical methods like membrane filters, micro filters, ultra filters, nano filters and reverse osmosis was successful in TDS reduction but caused mechanical problem. Therefore, integrated approach of biological and physical can solved the problem of TDS and reduces the operating cost considerably.

The good results were obtained from 2.5 N H_3PO_4 almond activated carbon in acid dye and 2.5 N H_2SO_4 almond activated carbon in metal complex dye.

CHAPTER - V

SUMMARY AND CONCLUSION

Textile dyeing industries are the one of the most polluted industries which creates lots of problems for environmental pollution. The textile dyeing industry produces in large amount of production and release of waste water effluent. Wastewater disposal may cause damage to the quality of the receiving water bodies, the aquatic eco-system, and the biodiversity of the environment. The removal of color from dye effluent, activated carbons is widely used as an adsorbent in the separation and purification of gas and liquid having potential applications in the textile industry. The present wastewater treatment schemes on the site of the plant consist of screening, pre-neutralization, anaerobic lagoon, post-neutralization, activated sludge process, and sedimentations.

In this study different types of oil cakes such as Almond, Sesame, Mustard, Coconut, Rice Bran, Cotton, Soya, Neem, Peanut, and Boiler ash were used for the preparation of activated carbon. And it was compared with commercial activated carbon. Six activated carbons were successfully prepared by carbonization and by the activation process. Oil cakes are the residue remaining after any oilseed has been pressed to remove the vegetable oil; it is used with other ingredients, as used animal foods. A cake made with vegetable oil instead of butter.

The oil cakes such as Almond, Rice bran, Sesame, Coconut, and Mustard, activated carbon was prepared by physical activation at 400˚C, followed by chemical activation by impregnating 2.5 N H_3PO_4, 1 N H_3PO_4, and 2.5 N H_2SO_4 in 1:5 ratios for overnight and is followed by physical activation at 300˚C for 2 hrs in a muffle furnace. The particle size of the activated carbon was further reduced by ball milling in Pulverisette 6 classic line (Fritsch, Russia) for 1 hour.

The textile acid dyes are used to dye natural protein fibers such as wool, silk, and also on nylon, and modified acrylics. An acid dye is a dye that is applied to textile materials at low pH. They contain sulphonic acid groups, which are usually present as sodium sulphonate salts. These increases solubility in water and give the

dye molecules a negatively charge. Acid dyes affix to fibers by hydrogen bonding, Van der Waals forces, and ionic bonding.

The metal complex dyes are also called metalized dyes in which one or two dye molecules are co-ordinated with a metal ion. The dye molecule is typically a mono structure containing additional groups such as hydroxyl, carboxyl, or amino. These are capable of forming a strong coordination complex with metal ions such as chromium, cobalt, nickel, and copper.

Considering the importance of the removal of dye effluent, research work entitled "Removal of residual Textile dye effluent using activated carbon prepared from agricultural residues" has been planned with the following objectives:

1. Selection of dye effluent
2. Selection and preparation of adsorbents
3. Characterization of adsorbents
4. Standardization process of color removal

METHODOLOGY

The study was conducted in the Department of Textile and Apparel Designing, Faculty of Community Science, Assam Agricultural University, Jorhat, Assam and Department of Textile Manufacturing and Textile Chemistry, Central Sheep and Wool Research Institute, Avikanagar, Rajasthan.

Keeping in mind the objectives of the study, the results obtained during the course of the investigation are summarized below:

- **Characterization of 2.5 N H_3PO_4 Almond activated carbon**

The good bulk density (4.533g/cm^3), porosity (4.500%), ash content (0.176%), moisture content (0.040%), methylene blue (123.667mg/g), pH (6.180), zero point charge (4.140pzc), particle size analysis (310.333nm) and iodine number (22.067m^2/g) were recorded in 2.5 N H_3PO_4 almond (ball milling) activated carbon. The adsorbent was found to be non-significant on iodine number.

- **Characterization of 1 N H_3PO_4 Coconut activated carbon**

The good bulk density (4.800g/cm^3), porosity (4.233%), ash content (0.218%), moisture content (0.063%), methylene blue (164.000mg/g), pH (5.660), zero point charge (3.850pzc), particle size analysis (112.333nm) and iodine number

(16.500m^2/g) were recorded in 1 N H_3PO_4 coconut (ball milling) activated carbon. The adsorbent was found to be non-significant on iodine number.

- **Characterization of 2.5 N H_3PO_4 Mustard adsorbent**

The good bulk density (4.567g/cm^3), porosity (4.567%), ash content (0.213%), moisture content (0.082%), methylene blue (125.333mg/g), pH (6.100), zero point charge (3.647pzc), particle size analysis (142.333nm) and iodine number (14.733m^2/g) were recorded in 2.5 N H_3PO_4 mustard (ball milling) activated carbon. The iodine number was found to be non-significant on adsorbent.

- **Characterization of 2.5 N H_2SO_4 Almond adsorbent**

The good bulk density (5.233g/cm^3), porosity (3.633%), ash content (0.172%), moisture content (0.071%), methylene blue (116.000mg/g), pH (6.973), zero point charge (6.460pzc), particle size analysis (825.000nm) and iodine number (21.5333m^2/g) were recorded in 2.5 N H_2SO_4 almond (ball milling). The adsorbent were found to be non-significant on ash content and iodine number.

- **Characterization of 2.5 N H_3PO_4 Rice Bran adsorbent**

The good bulk density (4.400g/cm^3), porosity (3.467%), ash content (0.160%), moisture content (0.056%), methylene blue (56.000mg/g), pH 6.997, zero point charge (3.913pzc), particle size analysis (390.333nm) and iodine number (16.100m^2/g) were recorded in 2.5 N H_3PO_4 rice bran (ball milling). The iodine number was found to be non-significant on adsorbent.

- **Characterization of 2.5 N H_2SO_4 Sesame adsorbent**

- The good bulk density (4.600g/cm^3), porosity (3.600%), ash content (0.160%), moisture content (0.033%), methylene blue (88.000mg/g), pH (6.527), zero point charge (5.643pzc), particle size analysis (1326.667nm) and iodine number (16.400m^2/g) were recorded in 2.5 N H_2SO_4 sesame (ball milling). The adsorbent were found to be non-significant on zero point charge and iodine number.

- **Characterization of commercial activated carbon**

The good bulk density (6.467g/cm^3), porosity (6.533%), ash content (0.118%), moisture content (0.117%), methylene blue (488.000mg/g), pH (5.343), zero point charge (5.750pzc), particle size analysis (447.667nm) and iodine number

(10.000m^2/g) were recorded in commercial activated carbon. The iodine number was found to be non-significant on adsorbent.

- **SEM (Scanning electron micrograph images) analysis of adsorbents**

The scanning electron micrographs of 2.5 N H_2SO_4 and 2.5 N H_3PO_4 of treated almonds were analyzed. Compared to 2.5 N H_2SO_4 treatments, the pores produced by the 2.5 N H_3PO_4 treatments were found to be smaller in size as compared to 2.5 N H_2SO_4 treatments. It was confirmed that the number of pores per unit area produced by H_3PO_4 treatment was high

EDX analysis of activated carbon

The EDX analysis of activated carbon prepared from the chemically treated 2.5 N H_3PO_4 and 2.5 N H_2SO_4 almond adsorbents showed that there was no significant change in the carbon content of the activated carbons prepared by the activation of mineral acids, however; the carbon content was seemed to be slightly higher in phosphoric acid treated with 2.5 N H_2SO_4 almond activated carbons. A negligible quantity of Na, K, S and Mg, Si, P, C elements were observed in case of 2.5 N H_3PO_4 and 2.5 N H_2SO_4 almonds activated carbons respectively.

Chemical Oxygen Demand (COD)and Biological Oxygen Demand (BOD): - A positive result was obtained in both the acid and metal complex dyes treated with 2.5 N H_3PO_4 almond than 2.5 N H_2SO_4 almond activated carbon.

XRD analysis of activated carbon: - The X-ray diffraction pattern of the 2.5 N H_2SO_4 almond and 2.5 N H_3PO_4 almond were recorded at peak 25˚ and 29˚ respectively, and indicated the presence of amorphous structure of the activated carbon with the diffraction pattern of (002).

FTIR analysis:- In our present study, the FTIR analysis result showed diverse types of functional groups such as O–H, C, O, C-C and C–O against the spectral peak.

BET analysis of activated carbon:- In BET analysis; the surface area, pore radius and pore volume of 2.5 N H_3PO_4 was recorded as 16.14 m^2/g, 0.85 nm and 0.029 cc/g and in 2.5 N H_2SO_4 almond, it was recorded as 64.28 m^2/g, 0.72 nm and 0.040 cc/g respectively.

- **Absorbency of Acid dye effluent:**

- **Percentage of absorbency 2.5 N H_3PO_4 Almond activated carbon**

In 2.5 N H_3PO_4 almond (ball milling) activated carbon, the lowest absorbency (1.120) was recorded in 0.5% concentration at 90 minute time period while the absorbency (0.840) was recorded in 1.0% concentration at 90 minute time period followed by (0.360) in 1.5% concentration at 90 minute time period and (0.107) in 2.0% concentration at 60 minute and 90 minute time period respectively. From the statistical analysis, it was clear that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 almond activated carbon.

- **Percentage of absorbency 1 N $_3PO_4$ Coconut activated carbon**

In 1 N H_3PO_4 coconut (ball milling) activated carbon, the lowest absorbency (1.223) at 0.5% concentration in 60 minute followed by (0.943) at 1.0% concentration in 90 minute, (0.500) at 1.5% concentration in 90 minute and (0.197) at 2.0% concentration in 90 minute of time period respectively. From the statistical analysis, it was clear that all the factors and their interactions were found to be significant at 5% level with respect to 1 N H_3PO_4 coconut activated carbon.

- **Percentage of absorbency 2.5 N H_3PO_4 Mustard activated carbon**

In 2.5 N H_3PO_4 mustard (ball milling) activated carbon the lowest absorbency were recorded (1.113) at 0.5% concentration in 90 minute followed by (0.850) at 1.0% concentration in 90 minute, while the absorbency (0.477) at 1.5% concentration in 90 minute and (0.133) at 2.0% concentration in 90 minute of time period. From the statistical analysis, it indicated that all the factors and their interactions were found to be significantly influence at 5% level with respect to 2.5 N H_3PO_4 mustard activated carbon.

➢ **Absorbency of Metal complex dye effluent**

- **Percentage of absorbency 2.5 N H_2SO_4 Almond activated carbon**

In 2.5 N H_2SO_4 almond (ball milling) activated carbon, the lowest absorbency were recorded (0.670) in 90 minute at 0.1% concentration while the absorbency (0.600) in 90 minute at 0.25% concentration followed by (0.267) in 30 minute at 0.5% concentration and (0.147) in 30 minute and 60 minute of contact of time period at 1.0% concentration respectively. From the statistical analysis, it indicated that all the factors and their interactions were found to be significantly influence at 5% level with respect to 2.5 N H_2SO_4 almond activated carbon.

- **Percentage of absorbency 2.5 N H_3PO_4 Rice Bran activated carbon**

In 2.5 N H_3PO_4 rice bran (ball milling) activated carbon, the lowest absorbency was recorded (0.533) at 0.1% concentration in 30 minute, (0.580) at 0.25% concentration in 30 minute, (0.487) at 0.5% concentration in 30 minute and (0.267) at 1.0% concentration in 60 minute of contact time period respectively. From the statistical analysis, it indicated that all the factors and their interactions except time, activated carbon*time, time*concentration and activated carbon* time*concentration were found to be non significant at 5% level with respect to 2.5 N H_3PO_4 rice bran activated carbon.

- **Percentage of absorbency 2.5 N H_2SO_4 Sesame activated carbon**

In 2.5 N H_2SO_4 sesame (ball milling) activated carbon, the lowest absorbency were recorded (0.620) at 0.1%concentration in 90 minute, (0.557) at 0.25% concentration in 60 minute, (0.357) at 0.5% concentration in 30 minute and (0.213) at 1.0% concentration in 30 minute of contact time period respectively. From the statistical analysis, it indicated that all the factors and their interactions except time and activated carbon*time were found to be non significant at 5% level with respect to 2.5 N H_2SO_4 sesame activated carbon.

➢ **pH of acid dye effluent**

- **pH of the effluent 2.5 N H_3PO_4 Almond activated carbon**

In 2.5 N H_3PO_4 almond (ball milling) activated carbon, it was recorded (4.523) at 2.0% concentration in 60 minute followed by (4.613) at 1.5% concentration in 90 minute while the pH (4.820) at 1.0% concentration in 60 minute and (5.120) at 0.5% concentration in 90 minute of contact time period respectively. From the statistical analysis, it indicated that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 almond activated carbon.

- **pH of the effluent 1 N H_3PO_4 Coconut activated carbon**

In 1 N H_3PO_4 coconut (ball milling) activated carbon, it was recorded (4.853), at 2.0% concentration in 60 minute, (4.893) at 1.5% concentration in 90 minute, (5.100) at 1.0% concentration in 60 minute and (5.330) at 0.5% concentration in 60 minute contact of time period respectively. From the statistical analysis, it indicated

that all the factors and their interactions were found to be significant at 5% level with respect to 1 N H_3PO_4 coconut activated carbon.

- **pH of the effluent 2.5 N H_3PO_4 Mustard activated carbon**

In 2.5 N H_3PO_4 mustard (ball milling) activated carbon, pH were recorded (4.530) at 2.0% concentration in 90 minute, (4.670) at 1.5% concentration in 30 minute, (4.863) at 1.0% concentration in 60 minute and (5.137) at 0.5% in 90 minutes of contact time period respectively. From the statistical analysis, it indicated that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 mustard activated carbon.

➢ **pH of Metal complex dye effluent**

- **pH of the effluent 2.5 N H_2SO_4 Almond activated carbon**

In 2.5 N H_2SO_4 almond (ball milling) activated carbon, it was recorded (5.233) in 90 minute at 1.0% concentration, (5.140) in 90 minute at 0.5% concentration, (5.163) in 30 minute at 0.25% concentration and (5.143) in 30 minute of contact time period at 0.1% concentration. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_2SO_4 almond activated carbon.

- **pH of the effluent 2.5 N H_3PO_4 Rice Bran activated carbon**

In case of 2.5 N H_3PO_4 rice bran (ball milling) activated carbon, it was recorded (4.773) at 1.0% concentration in 90 minute, (4.933) at 0.5% concentration in 60 minute, (5.017) at 0.25% concentration in 90 minute and (5.017) at 0.1% concentration in 90 minute of contact time period respectively. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 rice bran activated carbon.

- **pH of the effluent 2.5 N H_2SO_4 Sesame activated carbon**

In 2.5 N H_2SO_4 sesame (ball milling) activated carbon, it was recorded (5.153) at 1.0% concentration in 60 minute, (5.140) at 0.5% concentration in 60 minute, (5.193) at 0.25% concentration in 60 minute and (5.073) at 0.1% concentration in 30 minute of contact time period respectively. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_2SO_4 sesame activated carbon.

- **TDS of acid dye effluent**

- **TDS of the effluent 2.5 N H_3PO_4 Almond activated carbon**

In 2.5 N H_3PO_4 almond (ball milling) activated carbon, it was recorded as (2606.33mg/l) at 2.0% concentration in 30 minute followed by (2599.6mg/l) at 1.5% concentration in 30 minute, (2546.66mg/l) at 1.0% concentration in 30 minute and (2689.00mg/l) at 0.5% concentration in 90 minute of contact time period respectively. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 almond activated carbon.

- **TDS of the effluent 1 N H_3PO_4 Coconut activated carbon**

In 1 N H_3PO_4 coconut (ball milling) activated carbon, TDS were recorded as (2608.66mg/l) at 2.0% concentration in 90 minute, (2599.33mg/l) at 1.5% concentration in 90 minute, (2539.33mg/l) at 1.0% concentration in 60 minute and (2591.33mg/l) at 0.5% concentration in 30 minute of contact time period respectively. From the statistical analysis, it revealed that all the factors and their interactions except time, activated carbon*time, activated carbon* concentration and activated carbon* time*concentration were found to be non significant at 5% level with respect to 1 N H_3PO_4 coconut activated carbon.

- **TDS of the effluent 2.5 N H_3PO_4 Mustard activated carbon**

In 2.5 N H_3PO_4 mustard (ball milling) activated carbon, TDS were recorded as (2629.33mg/l) at 2.0% concentration in 30 minute followed by (2618.00mg/l) at 1.5% concentration in 60 minute, (2568.66mg/l) at 1.0% concentration in 30 minute and (2638.66mg/l) at 0.5% concentration in 30 minute and 60 minute of contact time period respectively. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_3PO_4 mustard activated carbon.

TDS of metal complex dye effluent

- **TDS of the effluent 2.5 N H_2SO_4 Almond activated carbon**

\In 2.5 N H_2SO_4 almond (ball milling) activated carbon, TDS was recorded (2110.00mg/l) at 1.0% concentration in 90 minute, (1138.66mg/l) at 0.5% concentration in 90 minute, (1889.33mg/l) at 0.25% concentration in 60 minute and

90 minute and (1549.33mg/l) at 0.1% concentration in 90 minute of contact time period respectively. From the statistical analysis, it revealed that all the factors and their interactions except activated carbon and time were found to be non- significant at 5% level with respect to 2.5 N H_2SO_4 almond activated carbon.

- **TDS of the effluent 2.5 N H_3PO_4 Rice Bran activated carbon**

In 2.5 N H_3PO_4 rice bran (ball milling) activated carbon, TDS was recorded (1889.33mg/l) at 1.0% concentration in 90 minute, (1939.33mg/l) at 0.5% concentration in 60 minute, (1959.00mg/l) at 0.25% concentration in 30 minute and (2047.33mg/l) at 0.1% concentration in 90 minute of contact time period. From the statistical analysis, it revealed that all the factors and their interactions were found to be non- significant at 5% level with respect to 2.5 N H_3PO_4 rice bran activated carbon.

- **TDS of the effluent 2.5 N H_2SO_4 Sesame activated carbon**

In 2.5 N H_2SO_4 sesame (ball milling) activated carbon, TDS were recorded as (2319.66mg/l) at 1.0% concentration in 60 minute, (2309.33mg/l) at 0.5% concentration in 30 minute, (2179.33mg/l) at 0.25% concentration in 30 minute and (2099.33mg/l) at 0.1% concentration in 90 minute of contact time period. From the statistical analysis, it revealed that all the factors and their interactions were found to be significant at 5% level with respect to 2.5 N H_2SO_4 sesame activated carbon.

CONCLUSION

From the study, it may be concluded that activated carbon is effectively engaged to remove the residual color from the textile dye effluent. The activated carbon production of agricultural by-products has potential economic and environmental impacts. In this study different types of oil cakes such as Almond, Sesame, Mustard, Coconut, Rice Bran, Cotton, Soya, Neem, Peanut, and Boiler ash were used for the preparation of activated carbons and it was compared with commercial activated carbon. Six activated carbons were successfully prepared by carbonization and by the activation process. The activated carbons prepared from oilcakes and are used in removal of color for acid and metal complex dye from wool dye effluent collected from the pilot plant. Sulphuric acid and Phosphoric acid treatment followed by low temperature pyrolysis converted the oilcakes into activated carbon. The pores size was reduced in the carbon color removal for dye adsorption.

The 92% color removal efficiency was found to be in 2.5 N H_3PO_4 almond in acid dye and 2.5 N H_2SO_4 almond in metal complex dye effluent. The synthesized activated carbon showed a lesser performance than commercial activated carbon under a low temperature of pyrolysis. Therefore, 2.5 N H_3PO_4 almond (ball milling) activated carbon acid dye and 2.5 N H_2SO_4 almond (ball milling) activated carbon metal complex dye effluent proved to be an excellent oil cake for preparation of activated carbon in the textile dye industry and can be used as an alternative source for wastewater treatment.

RECOMMENDATIONS

1. Different types of other agricultural residues can be used as wastewater treatment with different chemical activation.
2. Further studies can be done on reactive dyes for wastewater effluent treatment.
3. 2.5 N H_3PO_4 almond for acid dye and 2.5 N H_2SO_4 almond for metal complex dye can be used for textile dye effluent.

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Appendix

Table of FTIR spectra of activated carbon

S. No.	Wave length(cm^{-1})	Functional group
1.	2800–3200 cm^{-1}	O-H
2.	1650–1700 cm^{-1}	C-O and C=O
3.	3200–3600 cm^{-1}	O-H vibrations
4.	1100–1700 cm^{-1}	C-O and COOH
5.	3000–3100 cm^{-1}	Alkene group
6.	3500 - 3200 cm^{-1}	Hydroxyl, O-H functional groups
7.	1820-1600 cm^{-1}	C = O group
8.	1431.18 cm^{-1}	C = C bond
9.	1500-1400 cm^{-1}	C = C group
10.	1253.73 cm^{-1}	C-N groups
11.	1431.18 cm^{-1}	N = O
12.	3448 cm^{-1}	O-H stretching vibrations
13.	2924-2854 cm^{-1}	C-H stretching
14.	1419-1373 cm^{-1}	C-H stretching and symmetric C-H bending
15.	1597 cm^{-1}	C=C in the aromatic rings.
16.	1419 cm^{-1}	C-H bending in alkane or alkyl groups
17.	1267-1187 cm^{-1}	C-OH stretching vibrations in the carboxylic, phenolic or lactonic groups indicating the presence of oxygen functional group
18.	655 – 802 cm^{-1}	C – C stretching
19.	2800 - 2980 cm^{-1}	(C-H aliphatic stretching)
20.	1375 - 1465 cm^{-1}	(C-H aliphatic bending
21.	1655 cm^{-1} and 1703 cm^{-1}	Phenolic esters, carboxylic acid and conjugated ketonic structures
22.	3445 cm^{-1}	Due to O-H stretching
23.	2852 $cm^{-1}$and 2920 cm^{-1}	C–H stretching
24.	2374 cm^{-1}	C-C stretching
25.	1564 cm^{-1}	Stretching vibrations of C=C in an aromatic ring
26.	1630.5 cm^{-1}	C=O stretching
27.	1067.5 cm^{-1}	Stretching vibration of C-N and represents the S=O stretching.
28.	475.8 cm^{-1}	Fe-O stretching vibration
29.	188-1540-cm^{-1}	C=O stretching mode in carbonyls, carboxylic acids, and lactones