

**MICROENCAPSULATION OF ESSENTIAL OIL
ON
COTTON**

BY

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(2010HS169M)**

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2012

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This is to certify that this thesis entitled, “**Microencapsulation of Essential Oil on Cotton**”, submitted for the degree of **Master of Science**, in the subject of **Textile and Apparel Designing** to the CCS Haryana Agricultural University, is a bonafide research work carried out by **Latika Bhatt** Admission No. **2010HS169M** under my supervision and that no part of this dissertation has been submitted for any other degree.

The assistance and help received during the course of investigation have been fully acknowledged.

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With growing health care cost and sometimes impersonal quality of conventional medicine, consumers have turned to nature to find the answers to their questions. It has been realized that one should take personal responsibility of their health and strive to educate themselves on living more balanced lives. Therapies and medicines that were once viewed as alternative, cloaked in a shroud of scepticism, have risen from the shadows, providing a complement to conventional medicine. Aromatherapy is one such therapy which is a very powerful complementary therapy widely practiced today.

Aromatherapy has entered into the lifestyle of many consumers as a well-being solution to the stressful life in the global village. The term aromatherapy was coined in the late 1920s by the French cosmetic chemist R.M. Gattefosse, who noticed the excellent antiseptic properties and skin permeability of essential oils. Aromatherapy utilizes the controlled release of aromas or fragrances to promote feelings of comfort and well being amongst users. Aromatherapy is a form of alternative medicine in which healing effects are ascribed to the aromatic compounds in essential oils and other plant extracts (Wang and Chen 2005). Many common essential oils have medicinal properties that have been applied in folk medicine since ancient times and are still widely used today. Many are also claimed to have an uplifting effect on the mind.

An essential oil is a concentrated hydrophobic liquid containing volatile aroma compounds extracted from plants. Essential oils are also known as volatile oils, ethereal oils or aetherolea, or simply as the oil of the plant from which they were extracted, such as oil of clove. An oil is essential in the sense that it carries a distinctive scent, or essence, of the plant. Various essential oils have been used medicinally at different periods in history. Medical applications proposed by those who sell medicinal oils range from skin treatments to remedies for cancer, and often are based solely on historical accounts of use of essential oils for these purposes. Interest in essential oils have revived in recent decades with the popularity of aromatherapy, a branch of alternative medicine which claims that essential oils and other aromatic compounds have curative effects (Anonymous, 2012b). Oils are volatilized or diluted in carrier oil and used in massage, diffused in the air by a nebulizer, heated over a candle flame or burned as incense. Studies indicate that aromatherapy is effective for conditions such as anxiety, depression and boosting cellular immune functions. In many of the studies reviewed, scientists are suggesting further research for possible uses of essential oils as an alternative or complement to conventional medical practices (Michelle, 2010). Research studies have confirmed that both men and women are excited by the concept of

well-being benefits in clothes, especially those worn close to the body. A number of manufacturers and chemical companies have started offering range of health giving finishes and textiles.

Garments act as second skin hence are the excellent media to transfer the therapeutic effect of aromas to the human body and provide wellness effect through aromatherapy. The addition of fragrance to textiles has been carried out for many years in the form of fabric conditioners in the wash and tumble drying process but the effect was short lived. To achieve durable aroma finish on textiles has been a long time dream for textile chemists. As fragrances are volatile so they evaporate away and do not stay for long periods and are not resistant to wash. Therefore microencapsulation was found to be a solution to this problem. Microencapsulation can effectively control the release rate of fragrance compounds and essential oils as required which ensures the storage life of volatile substance (Slavica *et al.*, 2005).

Microencapsulation is a technique to prepare microcapsules, small particles that contain an active agent or core material surrounded by coating or shell of limited permeability (Benita, 1996). Microencapsulation is an innovation wherein active material is enveloped in a coating or capsule which protects it from external environment. The aroma capsule is a kind of container that provides both space for storing a certain amount of aromatic matter and a protective shield from the effects of sunlight and moisture. Release of aroma is by diffusion of the fragrance through the capsule wall and rupture of the capsules (Nelson, 2002). These capsules can be applied to fabric through simple pad dry sequence. During wear, simple mechanical rubbing of fabric gradually ruptures the membrane releasing active agent for therapeutic, energy boosting, stress busting, moisturising or deodorising effects.

As close friends of humans, textiles can make aromatherapy easy wherever they are needed. Various products such as fibres, fabrics, non-fabrics and garments can be chosen to enjoy the pharmaceutical and emotional effects of aromatherapeutic textiles. The uses of aroma textile are diverse. Interior textiles such as sheets, quilt-covers, curtains, carpets and bed-gowns are suitable for the attachment of lavender, camomile, citrus or cinnamon microcapsules, which are good for hypogenesis and eliminating fatigue. Patients suffering from high blood pressure felt sedation when they used pillow made of fabric treated with lavender, lemongrass, basil, rose or jasmine oil, which might have improved their work efficiency improved (Agrawal and Goel, 2010). The wellness business is proactive. People voluntarily become consumers to feel healthier, to reduce the effects of ageing and to avoid becoming consumers of the sickness business. Aromatherapeutic textiles offer a new challenge and field is wide open to innovation. It is believed that these textiles will become a fashion in the near future. This will not only provide health benefits to the common masses but will benefit the agriculture sector as it will ensure increased cultivation of aromatic plants

and enhanced rate of essential oil extraction. Aroma finish will add value to cotton so the cultivation, production and export of cotton will also boost.

So in order to exploit the therapeutic properties of aromatic oils and their application in textiles the present research is proposed with the following objectives:

1. To prepare microcapsules using essential oil and to standardise the process of encapsulation.
2. To study the wash durability of treated fabrics
3. To study the physical properties of treated fabrics.

CHAPTER-II

REVIEW OF LITERATURE

The review of literature reveals that not much has been conducted on microencapsulation with essential oils in India and abroad. An attempt has been made here to present a brief resume of the available literature on the issues relevant to the present study under the following sections:

- 2.1 Essential oils and aromatherapy
- 2.2 Microencapsulation technology
- 2.3 Related studies

2.1 Essential oils and aromatherapy

Gupta and Gupta, 2010 opined that aromatherapy has entered into the lifestyle of many consumers as a well-being solution to the stressful life of the global village in which we all live. Aromatherapy utilises the controlled release of aromas or fragrances of essential oils to promote feelings of comfort and well-being amongst users.

Agrawal and Goel, 2010 defined aromatherapy as the treatment or prevention of disease by use of essential oils. Two basic mechanisms were offered to explain the purported effects. One is the influence of aroma on the brain, especially the limbic system through the olfactory system. The other is the direct pharmacological effects of the essential oils. Essential oils can produce individual emotional responses when inhaled then noted by the brain. These responses can bring forth feelings of happiness, well being and peace hence the effect on the brain is psychological. These great health benefits can be attained in a safe, natural, drug-free way. They also revealed that aromatherapy does not interfere with any medications, it is simple and easy to use and the benefits are numerous. The most basic and most popular uses for aromatherapy include relaxation, calming, sedation and stimulation.

Fragrances are known to provide a broad spectrum of advantages when applied to performance apparel. Peppermint has an uplifting/head clearing and even muscle-easing effect which could be used for active sportswear, while lavender applied to bed sheets or pillow covers can relax and encourage sleep. Clear thinking/confidence building fragrances like jasmine and rose could also find use in women's formal wear (Gupta and Gupta, 2010).

In recent years, textile materials have been found in applications in the aromatherapy. A new sector of aroma therapeutic textiles is introduced and several commercial aroma therapeutic textile products are currently available in the market. On contact with human body and skin, aroma therapeutic textiles are designed to transfer an active substance for therapeutic purposes. The sedative and pharmaceutical effects of some essential oils have been listed below in Table 1 and 2 (Wang and Chen, 2005).

Table 1 : Sedative/Well-being effects of some essential oils

Emotions	Essential oils with sedative effects
Anxiety	Lemongrass, Chamomile, Rose, Cardamom, Clove, Jasmine, Lament Rose
Stimulation	Camphor, Balm oil
Anger	Chamomile, Balm oil, Rose,
Wretchedness	Basil, Cypress, Mint, Patchouli
Allergy	Chamomile, Jasmine, Balm oil
Distrustfulness	Lavender
Tension	Camphor, Cypress, Vanilla, Jasmine, Lemongrass, Sandalwood
Melancholy	Basil, Lemon, Chamomile, Vanilla, Jasmine, Lavender, Mint, Rose
Irritability	Chamomile, Camphor, Cypress, Lavender, Lemongrass
Hysteria	Chamomile, Balm oil, Lavender, Jasmine, Mania Basil, Jasmine, Pine
Desolation	Jasmine, Pine, Patchouli, Rosemary

Table 2 : Pharmaceutical effects of some essential oils

Effects	Essential oils
Sedation	Mint, Onion, Lemon, Lemongrass, Metasequoia
Coalescence	Pine, Clove, lavender, Onion, Thyme
Diuresis	Pine, lavender Onion, Thyme, Fennel, Lemon, Metasequoia
Facilitating Menses	Pine, lavender, Mint, Rosemary, Thyme, Basil, Chamomile, Cinnamon, Lemongrass
Dismissing sputum	Onion, Citrus, Thyme, Chamomile
Allaying a fever	Ginger, Fennel, Chamomile, Lemon
Hypnogenesis	Lavender, Fennel, Lemon, Lemongrass
Curing Hypertension	Lavender, Oregano, Basil, Chamomile
good for stomach	Pine, Ginger, Clove, Mint, Onion, Citrus, Rosemary, Thyme, Fennel, Basil, Cinnamon
Losing weight	Onion, Cinnamon, Lemon
Promoting appetite	Clove lavender, Mint, Onion, Citrus, Rosemary, Fennel Basil, Chamomile, Cinnamon, Lemon, Metasequoia
Curing flu	Pine, lavender, Mint, Onion, Citrus, Rosemary, Thyme, Chamomile.

The burgeoning demand for olfactory stimulation in part reflects the role of odour in influencing emotional state and well-being. Scents can enhance mood, promote optimism, relax or stimulate, aid the recall of personal memories, and facilitate creative thinking (Ehrlichman and Bastone, 1992; Herz, 1998). The introduction of an orange odour into a dentist office, reduced anxiety and improved mood in female patients (Lehrner *et al.*, 2000). Odours also play a role in influencing preferences for people, objects, and places across many situations and social interactions (Pierce *et al.*, 2004).

Shah and Goel, 2010 designed and developed perfumed lingerie sets using microencapsulated fragrances. Four popularly used microencapsulated fragrances were applied on four popularly used fabrics and tested for aroma retention. It was further revealed

that aroma was retained even after 30 cycles of wash, rub and abrasion. The perfumed lingerie was accepted and appreciated by the consumers

Kaur and Grewal, 2011 developed aromatherapeutic textile products for joint pains. Fabrics were treated with eucalyptus and jasmine microcapsules and were used for the treatment of respondents with joint pain. Knee caps and gloves were made for the respondents out of ribbed cotton fabric. Respondents suffering from joint pain had undergone for the wear trial for 2 months to get the results through interview schedule. The results revealed that the developed products had a positive effect on the emotional state of respondents (85%), improved quality of night sleep (35%), reduced medicine intake (65%) and decreased the local application of gel (50%). It was further recommended that the aromatherapeutic textile products made with eco-friendly chemicals like betacyclodextrin as binder and chitosan as sheath with eucalyptus and jasmine essential oils in core can be used to for developing products for the treatment of joint pains. These products retained their fragrance for at least 20 washcycles under controlled conditions.

Lec and Lee, 2006 studied the effect of lavender aromatherapy on students suffering from insomnia and depression patients in women college students. The purpose of this study was to explore the effects of lavender fragrance on sleep and depression in women college students. 42 college girls who complained of insomnia were studied during a 4 week protocol. It was found through the interview method that aromatherapy had a positive impact on the girls, they felt more fresh and energetic after being exposed to the oil.

2.2 Microencapsulation technology

Micro-encapsulation can be defined as the process of surrounding or enveloping one substance within another substance on a very small scale, yielding capsules, ranging from less than one micron to several hundred microns in size, with many useful properties. Microencapsulation results in the formation of microcapsules. Microcapsules may be spherically shaped, with a continuous wall, surrounding the core or may be asymmetrically and variably shaped with a quantity of smaller droplets of core material embedded throughout the microcapsule. All three states of matter may be microencapsulated which allows liquid and gas phase materials to be handled more easily as solids (Birla *et al.*, 2010). The core contents were released under controlled conditions to suit a specific purpose (Mei, 1995; Nelson, 2001; Simon, 2006). Figure 1 shows the general structure of a microcapsule which generally consists of two major components:

- 1. Active ingredient:** An active ingredient is the substance that may be in a liquid or solid form. It also refers to the core contents, internal phases, encapsulations, payloads or fillers.
- 2. Wall Shell:** A polymer coating that surrounds the active ingredients which may also be called the wall, shell, external phase, membrane or matrix. It may be natural, semi-synthetic or synthetic polymer (Cheng *et al.*, 2008).

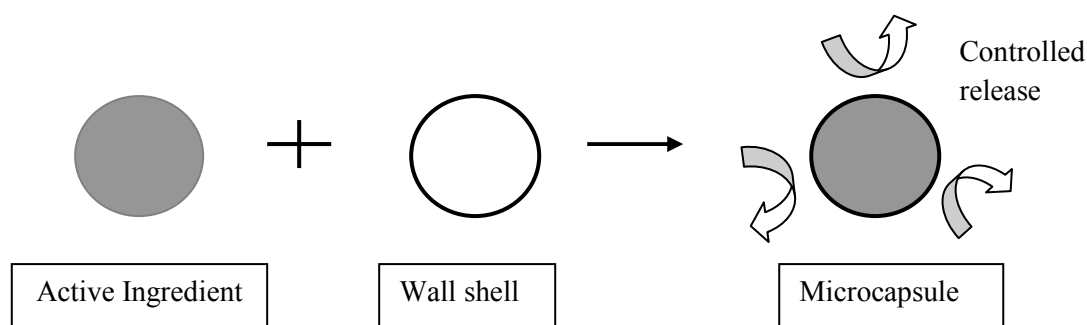


Fig. 1: General structure of microcapsule

The release mechanisms of the core contents vary depending on the selection of wall materials and more importantly, its specific end uses. Table 3 demonstrates the relationship between the textiles end uses and their release mechanisms. The core content may be released by friction, pressure, change of temperature, diffusion through the polymer wall, dissolution of the polymer wall coating, biodegradation etc (Anonymous, 2005; Holme,2003).

Table 3 : Textiles end uses and their release mechanism

End uses	Release mechanisms
Aromatherapy and Fragrance	Textiles Friction, Diffusion through Polymer Wall (Celessence International Limited, 2006)
Phase Change Material (Thermoregulation)	Temperature (Shin <i>et al.</i> , 2005)
Cosmetic Textiles (On contact with skin)	Friction, Pressure, Biodegradation (Achwal, 2003; Anonymous, 2005; Cognis, 2005)
Thermochromic and Photochromic (Colour Changing System)	Temperature, Ultra-violet Light (Sawada and Urakawa 2005; Sekar, 1998; Solar Active® International, Inc., 2007)
Flame Retardant Textiles Flame	High Temperature (Anonymous, 2005; Kover <i>et al.</i> , 1997)

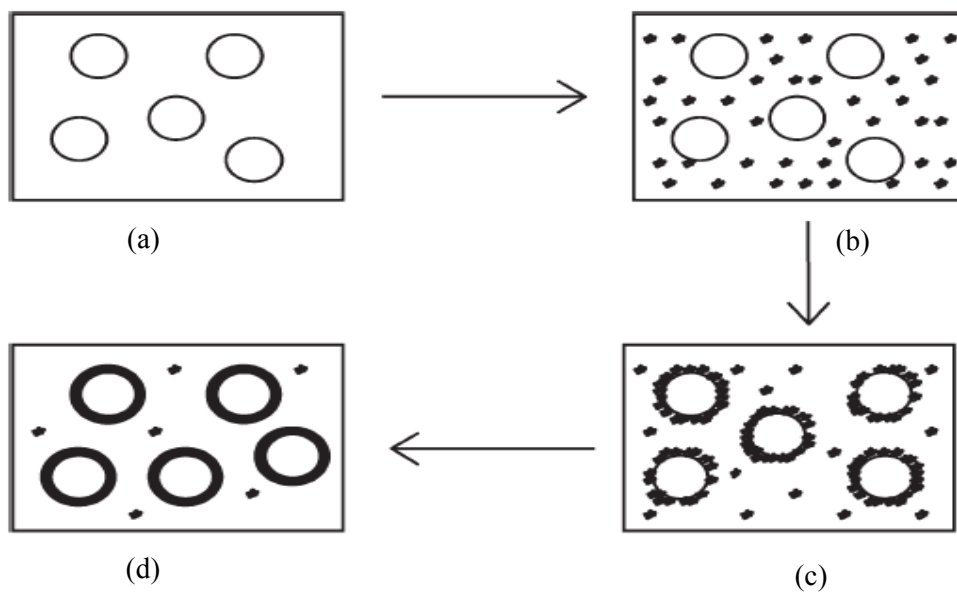
2.2.1 Techniques to prepare aroma microcapsules

Numerous preparation technologies available for the encapsulation of core material have been reported. The microencapsulation techniques that are more relevant for the encapsulation of aromatic/ essential oils are discussed below

a) Phase separation/coacervation : It is the approach of phase separation i.e. partial desolvation of a homogenous polymer solution into a polymer rich phase (coacervate) and the poor polymer phase (coacervation medium). Currently, two methods for coacervation are available, namely simple and complex processes. The mechanism of microcapsule formation for both processes is identical, except for the way in which the phase separation is carried out. In simple coacervation a desolvation agent is added for phase separation, whereas complex coacervation involves complexation between two oppositely charge polymers (Bansode *et al.*, 2010). The three basic steps in complex coacervation are: (i) formation of three immiscible phases; (ii) deposition of the coating (iii) rigidization of the coating. (Shin *et al.*, 2005)

Coacervation is a unique and promising microencapsulation technology because of very high payloads achievable and controlled release properties based on mechanical stress, temperature and sustained release. Coacervation is typically used to encapsulate oils (Korus, 2001; Korus *et al.*, 2003) but can also be adapted for the encapsulation of the nutrients, vitamins, preservatives, enzymes etc (Junyaprasert *et al.*, 2001).

The basic physical and polymer chemistry behind the coacervation process is well developed and understood (Dobetti and Pantaleo, 2002). The concept behind simple or complex coacervation microencapsulation is the phase separation of one or many hydrocolloids from the initial solution and the subsequent deposition of the newly formed coacervate phase around the active ingredient suspended in the same reaction media. The hydrocolloid shell can then be crosslinked, using an appropriate chemical or enzymatic crosslinker. A large number of hydrocolloid system have been evaluated for coacervation microencapsulation but the most studied and well understood system is the gelatine/ gum acacia system (Rabiskova and Malaskova, 1998 ; Jegat and Taverdet, 2001).



(a) core material dispersion in solution, (b) separation of coacervate from solution, (c) coating of core material by coacervate, (d) coacervate forms continuous shell around core

Fig. 2 : Schematic representation of coacervation process

b) Spray Drying: Microencapsulation by spray-drying is a low-cost commercial process which is mostly used for the encapsulation of fragrances, oils and flavours. Core particles are dispersed in a polymer solution and sprayed into a hot chamber. The shell material solidifies onto the core particles as the solvent evaporates such that the microcapsules obtained are of polynuclear or matrix type. Very often the encapsulated particles are aggregated and the use of large amounts of core material can lead to uncoated particles. However, higher loadings of core particles of up to 50–60% have been reported. Water-soluble polymers are mainly used as shell materials because solvent-borne systems produce unpleasant odours and environmental problems.

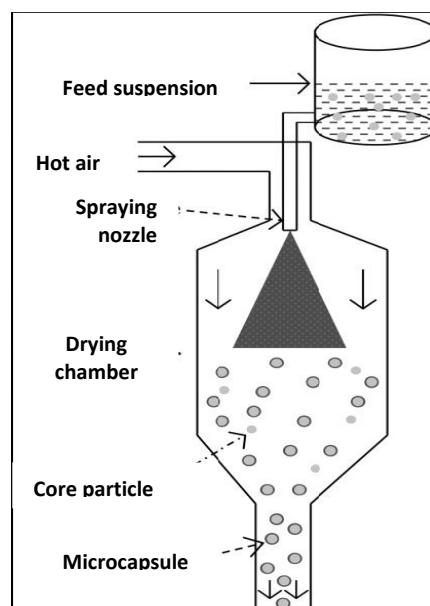


Fig. 3: Schematic diagram of spray-drying process

Schematic diagram (fig. 3) illustrating the process of micro-encapsulation by spray-drying (Birla *et al.* 2010).

c) Centrifugal Extrusion: Liquids are encapsulated using a rotating extrusion head containing concentric nozzles. In this process, a jet of core liquid is surrounded by a sheath of wall solution or melt. As the jet moves through the air, it breaks into droplets of core, each coated with the wall solution. While the droplets are in flight, a molten wall may be hardened or a solvent may be evaporated from the wall solution. Since most of the droplets are within $\pm 10\%$ of the mean diameter, they land in a narrow ring around the spray nozzle. Hence, if needed, the capsules can be hardened after formation by catching them in a ring-shaped hardening bath. This process is excellent for forming particles 400–2,000 μm in diameter. Since the drops are formed by the breakup of a liquid jet, the process is only suitable for liquid or slurry (Holme 2004).

Microcapsules can be applied to textiles by padding, coating, spraying or immersion without altering their feel or colour. For all these methods, a binder is required, it may be acrylic, polyurethane, silicon or starch. Its role is to fix microcapsules into the fabric and hold them in place during wash or mechanical agitation. The textile support may be cotton, silk, wool, nylon, polyester, leather, woven, knits, yarns and fibres. Also it can be applied to all type of clothing as well household linen (Marinkovic *et al.*, 2006).

2.3 Related Studies

Nelson, 2002a revealed that the contents of the capsules are released with light abrasion as would take place during day-to-day wear. He pointed out the fact that for the capsules to survive repeated washing and for application to fibres such as cotton,

microcapsules should be applied using a binder through padding, exhaust or screen printing techniques. A softener is also required, as unsoftened fabric containing microcapsules can sometimes appear to be stiffened.

Nelson, 2002b through his research revealed that the smaller capsules provide better covering of the product and the fragrance will last longer, as it takes longer for the capsules to be ruptured by physical pressure while larger capsules release more fragrance when ruptured but have a shorter life.

Korus *et al.*, 2003 opined that coacervation is a unique and promising micro-encapsulation technology because of very high payloads achievable and controlled release properties based on mechanical stress, temperature and sustained release. Coacervation is typically used to encapsulate oils

Zivdar and Najafi, 2004 studied the microencapsulation of liquid orange oil as a common flavoring agent in food industries by complex coacervation in a gelatin– gum Arabic polymeric wall system was studied. At a fixed ratio of 10% w/v as concentration of the materials used in this study, trend of changes of microencapsulation process variables using different wall polymeric contents along with varying levels of the core to wall ratio were investigated. Distribution pattern of the coacervate particle size showed that more than 70% of the particle with the average diameter of 9.68 mm were reasonably encapsulated in those treatments having core to wall ratio at the level of 1:1 and 1:2 while gelatin to gum arabic content of the wall system were set to be 1:1 and 2:1 ratio, respectively.

Wang *et al.*, 2005 developed aromatherapeutic textiles by using fragrance with β -cyclodextrin inclusion compounds and fixing them onto cotton fabrics with low temperature by using a conventional pad-thermofixed method. The fragrance release rates were greatly decreased and the results of sensorial evaluations showed that the performance of the fabric lasted for over 30 days.

Copete *et al.*, 2005 prepared chitosan-based microcapsules containing various active components and investigated their durability with a mixture of microcapsules and a binding agent. It was revealed that when microcapsules and binder are applied as a finish, the active ingredients were released and found to wash out less quickly, and a high degree of hydration was also achieved

Li *et al.*, 2005 investigated the effects of UV curing for encapsulated aroma finishing on cotton. The aroma function was prolonged to 50 wash cycles whereas the traditional curing method could only withstand 25 wash cycles. If a cotton fabric was finished with the selected aroma capsule and UV resin and cured under optimal conditions, the aroma function could withstand 50 wash cycles.

Lewis *et al.*, 2006 found that microencapsulated aroma can stay on textile for long periods since capsules greatly reduce the aroma evaporation rate. He used three fixing agents,

three kinds of thermal equipment and various curing conditions were used. It was reported that acrylic binder demonstrated excellent fixing of fragrance and the conventional hot air tenter and the infrared curing methods produced samples that could withstand at least 25 wash cycles.

Thilagavathi *et al.*, 2006 conducted a study on microencapsulation of herbal extracts such as *neem* and Mexican daisy using simple coacervation technique. The herbal extracts were used as core material and gum acacia as wall material and later applied it onto fabric using pad-dry-cure method. The light microscopy with image processing revealed the presence and alignment of microcapsules on the fabric. Durability test comparing microencapsulated and directly applied herbal extracts revealed that microencapsulated samples retained their activity for more than 15 wash cycles while the directly applied herbal extraction did not showed much activity after 10 washes, this was because the extracts were coated only on the surface.

Fabien *et al.*, 2009 noticed that it's not necessary to have a high concentration of binder and microcapsules to achieve the best washing fastness. Determine a good proportion of microcapsule and binder to limit the loss of coating weight after wash.

Sathianarayanan, 2009 conducted a research by treating cotton fabric with herbal extracts that act as bactericides as well bacteriostats. It was noticed that extracts applied directly in fabric through pad dry cure method were not durable to wash and can be used for the manufacture of disposable products but microencapsulated fabric showed durability to washing upto 15 wash cycles. The surface morphological data revealed that microencapsulated fabrics become stiff and their crease recovery property also decreases.

Shiqui *et al.*, 2009 studied the effect of finishing methods on microencapsulated aroma finishing. Lemongrass aroma microcapsules on 100% cotton woven fabric using DMDHEU, a polyurethane and acrylic binder. Fragrance durability of finished samples was examined by washing and odour testing. The results revealed that DMDHEU resin is not a good fixing agent for holding lemon fragrance capsules. It could withstand only 5 wash cycles if fabric was cured at 177°C for 1 min in a conventional hot air tenter. Polyurethane binder also lost their fragrance between 1 to 5 wash cycles but acrylic binder exhibited excellent wash durability i.e. 20 wash cycles.

Sukumar and Lakshmikantha, 2009 formulated cotton fabric with distilled jasmine and lavender oil as core material by employing the microencapsulation technique. The aromatic oils were microencapsulated using gelatine and sodium alginate as wall material by coacervation technique. These microcapsules were fixed in 100% cotton fabric with the binder by pad- thermo fixation. They further revealed that the capsules were of uniform size and retained finish till 25 wash cycles. It was also reported that aromas had good pharmacological effect on people.

Salaun *et al.*, 2009 revealed that the efficiency of a binder to link microcapsules on a textile surface depends on the compatibility of the different interfaces of the products involved in the coating process. The choice of the binder adapted to the microcapsules by the comparison of surface energy components induced by the contact angle measurement method and washing tests was studied. It was found that polyurethane- based binder was the most suitable to link melamine formaldehyde microcapsules. It was further revealed that the adhesion of microcapsules was closely dependent on the chemical nature and structure of the textile support.

Monllor and Sanchez, 2009 conducted a research to assess the presence of fragrance microcapsules on cotton fabric using different padding applications and evaluated them using scanning electron microscopy (SEM) and fourier transform infrared spectroscopy (FTIR). The ratios proposed showed that when the concentration of microcapsules in the padding bath increased, their value increased too. Secondly, he also analyzed the effect on thermal treatment on microcapsules was also analyzed. This was undertaken using hot air at 120°C, 140°C and 160°C, or by ironing the fabric impregnated with microcapsules at 110°C, 150°C and 200°C, by ironing 1, 5 and 10 times on the analyzed zone. It was found that when the temperature was higher than 120°C, microcapsules were deflated and damaged.

Agrawal and Goel, 2010 conducted a research on microencapsulation. Citronella oil was used as core material and gum acacia as wall material and optimised the process for different concentration and temperature. It was revealed that microcapsules were formed in two ratios only i.e. 1:2 and 2:1 and in both the cases the size varied. Both the larger and the smaller capsule have their own advantages. Further it was concluded that microencapsulation can effectively control the release rate of the fragrance compounds and essential oils as required, which ensures the storage life of volatile substances.

Gupta and Gupta, 2010 revealed that for most wellness finish on textiles use microencapsulation to deliver active ingredient like moisturizer, therapeutic oils or insecticides through the clothes onto skin directly where they are absorbed by the dermis.

Monllor and Sanchez, 2010 studied the influence of the resin on the stability of microcapsules to cotton fabric. A fixed quantity of microcapsules was established and the resin concentration was varied in the padding bath in order to evaluate the influence of the resin in order to make microcapsules remain on the fabric for greater length of time. The results revealed that without resin, microcapsules remained on fabric till 10 wash cycles but when 10g/l acrylic resin was used the microcapsules remained on fabric till 20 wash cycles. Resin quantities more than 10g/l were not recommended by commercial brands because it changed the fabric hand.

Danfeng *et al.*, 2012 prepared microcapsules by complex coacervation using gelatin and acacia as wall materials and olive oil as the core substance. Process parameters, such as

the dosage of the crosslinker, concentration of the wall materials, pH Value and the ratio between core and wall materials were analysed in detail. Moreover, crosslinking degree of wall materials was more important in obtaining good slow release microcapsules. The morphology and particle size distribution of the microcapsules were analysed by scanning electron microscope and laser particle size analyzer. The oil content and the release rate of the olive oil were also studied. In order to obtain microcapsules with good mobility and dispersal, a spray drying process was used to dry the product. The olive oil microcapsules were obtained with particle size of 3-8 micrometer and an oil content of about 60%. The process was optimized using 3 ml dosage of the cross linking agent, 3% concentration of wall materials , pH value of coacervation was 4.0 and 1:1 ratio of core/wall material. Olive oil microcapsules prepared with these optimal process conditions had good disperse effect and high encapsulation efficiency.

The present study was carried out to impart functional and aesthetic finish to cotton fabric using essential oil. The experimental procedure has been explained under the following sections:

- 3.1 Selection of material
- 3.2 Preparation of the fabric
- 3.3 Determination of preliminary data of the fabric
- 3.4 Selection of microencapsulation technique
- 3.5 Standardization of microencapsulation process
- 3.6 Standardization of padding process
- 3.7 Application of microcapsules on fabric
- 3.8 Assessment of microencapsulated fabrics for wash durability
- 3.9 Testing of physical properties
- 3.10 Analysis of data and application of statistical tools

3.1 Selection of Material

3.1.1 Selection of the fabric: A survey was conducted in the local market of Hisar, Haryana, to identify different types of cotton fabrics commonly used by consumers for apparels and a medium weight fine cotton fabric was selected and procured.

3.1.2 Selection of essential oil: For this study an exhaustive list of various essential oils was prepared out of which four oils were chosen on the basis of availability, cost and exclusion of essential oils on which similar researches have been carried out. Out of four one essential oil was selected with the help of a self structured preferential choice index on two parameters i.e. aroma of the oil and its therapeutic effects and was subjected to 20 experts. All features were assessed using a 5 quantum scale (Annexure-I).

Rating	Weightage
Most Preferred	5
Preferred	4
Moderately Preferred	3
Less Preferred	2
Not Preferred	1

3.1.3. Selection of wall material : Wall materials are used to encapsulate the core material. A natural gum which is compatible with cotton fabric and other ingredients of microencapsulation process was selected on the basis of reviews.

3.2 Preparation of Fabric

To prepare a textile material for padding, it must undergo preparation to ensure complete wetting, uniform absorbency of the microcapsules. Desizing, scouring and bleaching treatments were given to fabric to remove foreign material and for whitening of the fabric before imparting finish.

3.2.1 Desizing of cotton fabric: Desizing was done to remove starch and any other sizing material present in the fabric. The fabric was desized in a solution containing 1 percent sulphuric acid (H_2SO_4) at $50^{\circ}C$ with material liquor ratio 1:40 for 60 minutes. The fabric was rinsed thoroughly to remove loose starch and other residues, if left (Modi and Grade, 1975).

3.2.2 Scouring of cotton fabric: Scouring was done for the removal of natural and added impurities like oils, fats, waxes and other adventitious dirt that may have been added to the fabric during manufacturing process. The fabric was weighed and soaked prior to impregnating in scouring bath. The fabric was squeezed thoroughly and was treated in a solution containing 1 percent soap, 3 percent soda ash and 0.5 percent sodium sulphite at boiling temperature ($100^{\circ}C$) with material liquor ratio 1:40 for 60 minutes. After that the fabric was rinsed thoroughly to remove any residues, if left and dried on a flat surface (Modi and Grade, 1975).

3.2.3 Bleaching of cotton fabric: The bleaching process removes the natural colouring matter present in fabric and makes it more absorbent and whiter. Scoured cotton fabric was bleached in a solution containing 1 per cent hydrogen peroxide, 1 percent sodium silicate, 0.5 percent soda ash, 0.1 percent detergent and 1 percent sodium carbonate at $80-85^{\circ}C$ for 60 minutes with material to liquor ratio 1:40. The fabric was removed from solution, washed thoroughly and dried on a flat surface (Chackraverty and Trivedi, 1979).

3.3 Determination of Preliminary Data of Textile Material

The preliminary data of the fabric to be treated was taken under three parameters i.e. fabric count, weight and thickness. The fabric samples were conditioned prior to determination of fabric dimensions under standard test conditions.

3.3.1 Fabric count: The fabric count is the number of warp yarns (ends) and filling yarns (picks) per inch in a woven fabric. Paramount pick glass with pointer was used to determine fabric count using ASTM-D 123 test method. It was determined by counting the number of threads per square inch in the warp and weft directions at five different places in the fabric. An average of five readings was taken.

3.3.2 Fabric weight: The weight of fabric is defined as weight of a known area of the material and then computing the weight per unit area. Samples were cut at random from fabric with the help of round cutter for GSM. The samples were weighed separately on the Paramount Precision Scale for GSM (grams per square meter) using ASTM –D 3776-90 test method. An average of five readings was taken and weight per unit area in grams per square meter was calculated.

3.3.3 Fabric thickness: Fabric thickness is defined as ‘the distance between two parallel surfaces while exerting a specified pressure on the material’, the pressure to be applied on the specimen by the pressure foot of the tester. Paramount thickness tester was used to determine the thickness of fabrics using ASTM-D 1777-96 test method. A specimen was placed on flat surface below pressure foot of the instrument without any folds and wrinkles. The pressure foot was lowered upon the specimen gently until the pointer of the dial meter stopped moving further and the reading on the dial gauge was recorded in mm. An average of five readings was calculated as the fabric thickness.

3.4 Selection of Microencapsulation Technique

Out of the many physical and chemical techniques of microencapsulation i.e. solvent evaporation, polymerization, spray drying, pan coating, phase separation centrifugal extrusion etc. The phase separation complex coacervation technique was selected on the basis of review and the suitability of the process to be carried out in the laboratory of the department.

Complex coacervation technique: Gum was taken as the wall material and essential oil as the core material. Gelatin is the common ingredient in all the processes of complex coacervation. The basic recipe was followed for the formation of microcapsules but the expected results were not obtained. Hence modifications were made and various concentrations of the raw material were optimized. The basic recipe referred is as follows:

12 g of gelatin was accurately weighed and dissolved in 25 ml of warm water. This was stirred with high speed stirrer for 10 minutes. 4 g of core material was added to the solution at 40°C. 12 g of gum acacia was weighed accurately and dissolved with 25 ml warm water separately. The gum acacia solution was added to the gelatin solution and the temperature of the solution was maintained at 40°C. The pH of the solution was decreased to 4.5 by addition of dilute acetic acid. The solution was stirred at high speed for 20 minutes and the temperature was lowered to 5°C for gel formation. The pH was increased to 8.5 by addition of sodium hydroxide solution to form microcapsules. 1 ml of alcoholic formalin (17 percent) was added to the formed capsules for stabilization (Teli *et al.*, 2005).

3.5 Standardization of Microencapsulation Process

The different variables of microencapsulation process i.e. ratio of gum, oil and gelatin, temperature and pH were optimized one by one. The basic recipe was modified by incorporating different amount of gelatin, gum and oil, subjected to different range of temperature and pH and the resultant precipitate obtained after each process was analyzed under inverted microscope to ensure the formation of microcapsules and images were captured. The combinations of the ratio of gelatin, gum and oil which produced the desired results were further subjected to optimization of the other variables. At a time the ratio of only one ingredient was varied and other ingredients and variables were kept constant.

The process is illustrated stepwise in fig. 4

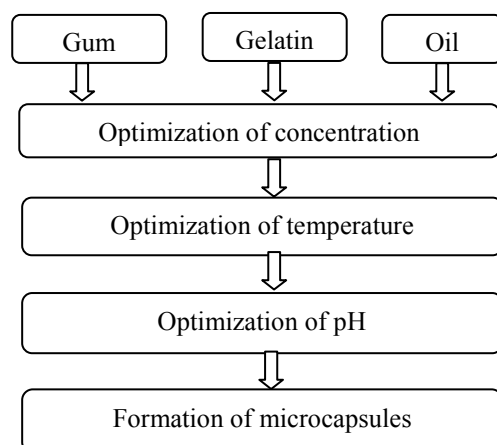


Fig. 4 : Flowchart of standardization of microencapsulation process (complex coacervation process)

3.5.1 Optimization of ratio of essential oil: To optimize ratio of essential oil, five different ratio of oil i.e. 0.5, 1.0, 2.0, 2.5 and 3.0 were taken while other raw material ratios and variables were kept constant and microencapsulation was carried out. The resultant precipitate obtained was analyzed under inverted microscope to ensure the formation of microcapsules and optimization was done on the basis of the visual assessment of the size of the microcapsules, uniformity in size and distribution and wall of the microcapsules on comparative basis. The one giving best results was selected for next stage of optimization.

3.5.2 Optimization of ratio of gum: For determination of optimum ratio of gum, five different ratio of gum i.e. 1, 2, 3, 4 and 5 were taken with optimized ratio of essential oil whereas ratio of gelatin was kept constant alongwith all other variables. Microencapsulation was carried out and ratio of gum was optimized on the basis of visual assessment as explained in section (3.5.1)

3.5.3 Optimization of ratio of gelatin: For determination of optimum ratio of gelatin, five different ratio of gelatin i.e. 1, 2, 3, 4 and 5 were taken with optimized ratio of oil and gum whereas all other variables were kept constant. Microencapsulation was carried out and ratio of gelatin was optimized on the basis of visual assessment as explained in section (3.5.1)

3.5.4 Optimization of temperature: For determination of optimum temperature for microencapsulation the process was carried out at five different temperature i.e. 30, 40, 50, 60 and 70°C with optimized ratio of gum, oil and gelatin whereas other variables were kept constant. Temperature was optimized on the basis of visual assessment as explained in section (3.5.1)

3.5.5 Optimization of initial and final pH: For optimization of pH, the optimized ratio of gum, essential oil and gelatin at optimized temperature was set to initial pH 4.0, 4.5, 5.0, 5.5, 6.0, 6.5 and 7.0. The microencapsulation process was carried out till gel formation took place and then final pH was maintained at 7.0, 7.5, 8.0, 8.5, 9.0, 9.5 and 10.0 with each initial pH.

The initial and final pH was optimized on the basis of visual assessment of microcapsule gel as explained in section (3.5.1)

Microcapsule gel was prepared using the standardized recipe. Optimized ratio of gelatin was dissolved in warm water and was stirred at high speed for 10 min. Optimized ratio of essential oil (core material) was added to the solution at optimized temperature and pH of the solution was set at optimized initial pH. The solution was stirred at high speed for 20 min and temperature was lowered to 5°C for gel formation. The pH of the gel was set at optimized final optimized pH. 1 ml of alcoholic formalin (17 percent) was added to the formed capsules and the formed microcapsule gel was stored for further application on fabric.

3.6 Standardization of Padding Process

The padding process was standardized for different variables like material to liquor ratio (MLR), proportions of microcapsules, binder and softener, curing time and temperature. The cotton fabric sample was immersed in the padding solution with 110-120 expression using pneumatic padding mangle, squeezed and cured at 80-85°C for few minutes (Sukumar and Lakshmikantha , 2009 and Thilagavathi et.al., 2006).

The resultant fabric obtained after each padding and curing process was analyzed under e-scope to ensure the presence of microcapsules in the fabric structure and images were captured.

The process is illustrated stepwise in Fig. 5

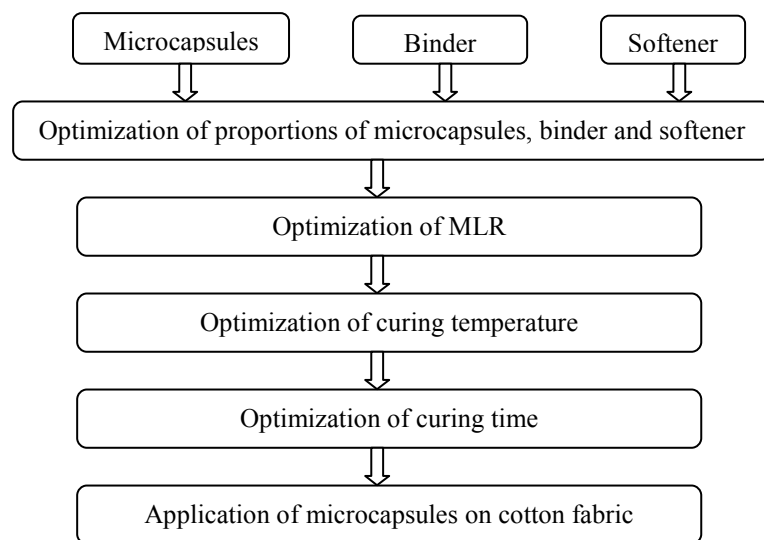


Fig. 5: Flowchart of standardization of padding process

3.6.1 Optimization of proportion of microcapsule gel in padding bath: To optimize the proportion of microcapsule gel, three different proportions of microcapsule gel i.e. 30, 40 and 50 (g/l) were taken while the proportion of binder, softener and other variables remained constant. The padding and curing of the fabric was carried out and the resultant fabric was analyzed under e-scope and optimum proportion was selected on the basis of visual analysis

for presence of microcapsules on fabric on comparative basis, wash durability, bending length and flexural rigidity.

3.6.2 Optimization of proportion of softener in padding bath: For determination of optimum proportion of softener, three different proportions of softener i.e. 1, 3 and 5 were taken with optimized proportion of microcapsule gel and binder proportion was kept constant alongwith all other variables. Padding and curing was carried out and optimization was done as explained in section (3.6.1)

3.6.3 Optimization of proportion of binder in padding bath: For determination of optimum proportion of binder, three different proportions of binder i.e. 10, 15 and 20 were taken with optimized proportion of microcapsule gel and softener and all other variables were kept constant. Padding and curing was carried out and optimization was done as explained in section (3.6.1)

3.6.4 Optimization of MLR: For determination of optimum material to liquor ratio, three different material to liquor ratio i.e. 1:20, 1:30, and 1:40 were taken with proportion of binder: softener: microcapsule gel whereas temperature and time were kept constant. Padding and curing was carried out and optimization was done as explained in section (3.6.1)

3.6.5 Optimization of curing temperature: For determination of optimum curing temperature, the process was subjected to three different temperatures i.e. 70, 80 and 90°C with optimized proportion of binder, softener and microcapsule gel using optimized MLR and time was kept constant. Padding and curing was carried out and optimization was done as explained in section (3.6.1)

3.6.6 Optimization of curing time: For determination of optimum curing time, the process was subjected to three different time i.e. 30, 60 and 90sec with optimized (binder, softener and microcapsule gel), MLR and curing temperature. Padding and curing was carried out and optimization was done as explained in section (3.6.1)

3.7. Application of Microcapsules on Fabric

The microcapsules formed by the optimized process through complex coacervation. The microcapsules were applied on cotton fabric with the help of padding mangle as explained in section (3.6) using the optimized recipe of padding and cured at optimized temperature and time.

3.8 Assessment of Microencapsulated Fabrics for Wash Durability

Wash durability test was carried out as per the recommendation of IS: 3361-1979 (ISI handbook of textile testing, 1982e) in a 'laundrometer'. Treated fabric was cut into sample size 10×4 cm. A soap solution was prepared using 5g of soap per litre of distilled water.. Each fabric sample was placed in a separate container and soap solution was added to each container maintaining M:L ratio 1:50. These specimens were treated for 45 minutes at 50 ± 2° C. After removing fabric specimen from laundrometer after 5, 10, 15, 20, 25 and 30 wash

cycles it was rinsed twice in cold water and then in running tap water for 10 min and squeezed. The finished specimen were dried in air at a temperature not exceeding 60 °C. These samples were further used for microscopic analysis and to check the retention and intensity of aroma.

3.8.1 Microscopic analysis of treated fabrics after wash: Scanning electron microscope was used to ensure the presence of microcapsules in the treated samples and samples after different number of wash cycles i.e. 5, 10, 15, 20, 25 and 30 as explained in section (3.8). The treated fabric samples of 10 mm diameter were mounted on the circular metallic sample holder and samples were analyzed at a magnification of 1200 x and images were captured.

3.8.2 Retention of aroma after wash: The treated samples and washed samples as explained in section (3.8) were evaluated against aroma retention by 20 experts through olfactory analysis. The aroma assessment was based on Lewis’s procedure (Lewis, 2003). A portion of each fabric was removed after 5, 10, 15, 20, 25 and 30 wash cycles, tested for the presence of fragrance by the experts. The washed samples were evaluated within 24-hr after washing. The samples were hung on a clothesline in a room for 1 hour to stabilize the evaporation of fragrance prior to being judged. To get fair judgement the experts were not allowed to enter the stabilizing room and samples were brought to the experts. To detect odor, a specimen was put on a desk and the expert used a fingernail to scratch “X” on the specimen to rupture some capsules and smell the swatch. The size of “X” was about 3x3 cm. The response were recorded as ‘Yes’ or ‘No’. If the aroma was judged to be present by majority of experts, the condition was noted as passing.

3.8.3 Intensity of aroma after wash: The treated samples and washed samples as explained in section (3.8) were evaluated qualitatively for intensity of aroma at different number of wash cycle. Change in amount of aroma was assessed by sensory evaluation of 20 respondents with the help of self structured criteria (Annexure II) and scores were assigned as follows :

Rating	Weightage
Very strong	5
Strong	4
Moderate	3
Mild	2
Faint	1

For each feature the frequency of each rating was multiplied by the respective weightage to calculate the weighted mean score.

The samples were finally rated on the basis of their weighted mean score as per following scale.

Weighted Mean Score (WMS)	Rating
4.2 – 5 .0	Very strong
3.4 - 4.2	Strong
2.6 - 3.4	Moderate
1.8 - 2.6	Mild
1 - 1.8	Faint

3.9 Testing of physical properties

Physical properties play an important role in the buying and selling of fabrics as well as in consumer's use. Assessment of physical properties aims to predict behaviour of fabric in use. It aids in defining the quality of a fabric or other properties associated with serviceability as there is not always, a clear dividing line between physical and performance properties. Various physical properties of controlled and treated fabrics were tested to analyze the effect of aroma finish and to compare the changes that have occurred after the treatment of finish, hence to ensure their suitability for their intended purpose and expected quality. The fabrics were compared by testing various physical properties like fabric count, weight, thickness, fabric stiffness or bending length , flexural rigidity, tensile strength and elongation.

3.9.1 Fabric count, weight and thickness: The fabric count, weight and thickness of the treated fabric was tested as per procedure mentioned in section (3.3.1), (3.3.2) and (3.3.3) respectively.

3.9.2 Bending length and flexural rigidity: Bending length is the length of fabric that will bend under its own weight to a definite extent. It determines the drapability of fabric. Bending length is related to the quality of stiffness that is appreciated by visual examination of the draped material, in the sense that clothes having a high bending length tend to drape stiffly.

The bending length of the fabrics was determined by the paramount stiffness tester using ASTM-D I388-64 test method. Samples of size 25 x 200 mm were cut from warp and weft directions with the aid of template and conditioned. Both template and sample were transferred to the platform with the fabric underneath, coinciding the zero mark of the scale and zero line engraved on the side of platform. The template was moved slowly over 41.5° sloped along with the strip till the top of the specimen viewed in the mirror cut both index lines. The bending length was read from the scale, which coincided with the front edge of the top plate. Each sample was tested four times in both warp and weft direction.

Reading from the scale was noted in mm and an average of four readings was calculated for both the directions. Weighed all the test samples and also measured their length and width. Calculated their areas and determined their weight per unit area in mg/sq. cm.

$$\text{Average Bending Length} = \frac{S}{4 \times n}$$

Where

S = Sum of total bending length of specimens (four readings per specimen in either warp or weft direction)

n = Total number of specimen tested in warp or weft direction.

Flexural rigidity:

Warp direction (G1)

$$G1 = W \times C1 \text{ mg-cm}$$

Weft direction (G2)

$$G2 = W \times C2 \text{ mg-cm}$$

Where,

W = Weight per unit area of the fabric in milligrams per square centimeter.

C = The mean bending length for the respective direction.

Overall flexural rigidity (G)

$$G = \sqrt{G1 \times G2}$$

In the same way bending length and flexural rigidity of all samples, treated and untreated were measured.

3.9.3 Tensile strength and elongation : Tensile strength is the ability of the fabric to withstand the load of force usually expressed as kilogram weight or pound weight and elongation of fabric corresponding to tensile strength is the original length of the sample at breaking point.

Tensile strength of fabrics was determined on paramount digital tensile strength tester for textiles (Analogue Model), using ASTM-D 5035 test method.

The sample of size $6 \times 4 \pm 0.05$ inches were cut from warp and weft direction of the fabric with the help of template. The samples were mounted between the jaws with approximately 1.5 inch of fabric protruding from each side of the jaws at a distance of 3 inches. The speed of upper jaw was adjusted at 300 ± 10 mm/min. The machine was started and the upper jaw moved in upward direction. The readings were taken from the digital display at sample break. The specimen should be slightly tensioned for getting accurate elongation at break. An average of 5 readings of the specimen from both directions (warp and weft) was taken and the average was calculated.

3.10 Analysis of Data and Application of Statistical Tools

The collected data were coded, tabulated and analyzed using suitable statistical tools to draw the meaningful inferences.

Frequency: Frequency was calculated and used to calculate percentage and weighted mean score

Percentages: Simple percentages were calculated for the analysis of data regarding retention of aroma after wash.

Weighted Mean Score: To quantify the data regarding preferential choice of essential oil, retention and intensity of aroma after wash, weighted mean score was calculated and ranks were allotted on that basis.

$$\text{Weighted Mean Score} = \frac{\text{Total weighted score}}{\text{No. of respondents}}$$

The present study was undertaken to encapsulate lemongrass oil and apply those microcapsules on cotton fabric to impart functional and aesthetic finish. The process of microencapsulation as well as the padding was standardized. Wash durability, aroma assessment and various physical properties were tested. The results obtained are explained under the following sections:

- 4.1 Preliminary data of the fabric
- 4.2 Selection of essential oil
- 4.3 Standardization of microencapsulation process
- 4.4 Standardization of padding process
- 4.5 Assessment of microencapsulated fabrics for wash durability
- 4.6 Effect of finish on physical properties of cotton fabric

4.1 Preliminary Data of the Fabric

Preliminary data of the selected cotton fabric was studied before applying microcapsules. The preliminary data of the fabric was further used to compare the physical properties of the controlled sample with the microencapsulated fabric. Table 1 indicates that the count of the fabric used for the study was 83×57 ends and picks per inch, weighing 37.33 gm/sq.mt. and thickness was 0.276 mm.

Table 1: Preliminary data of the fabric

Fabric	Fabric count (ends and picks/ inch)	Fabric weight (g/ sq. mt.)	Fabric thickness (mm)
Cotton	83 × 57	37.33	0.276

4.2 Selection of Essential Oil

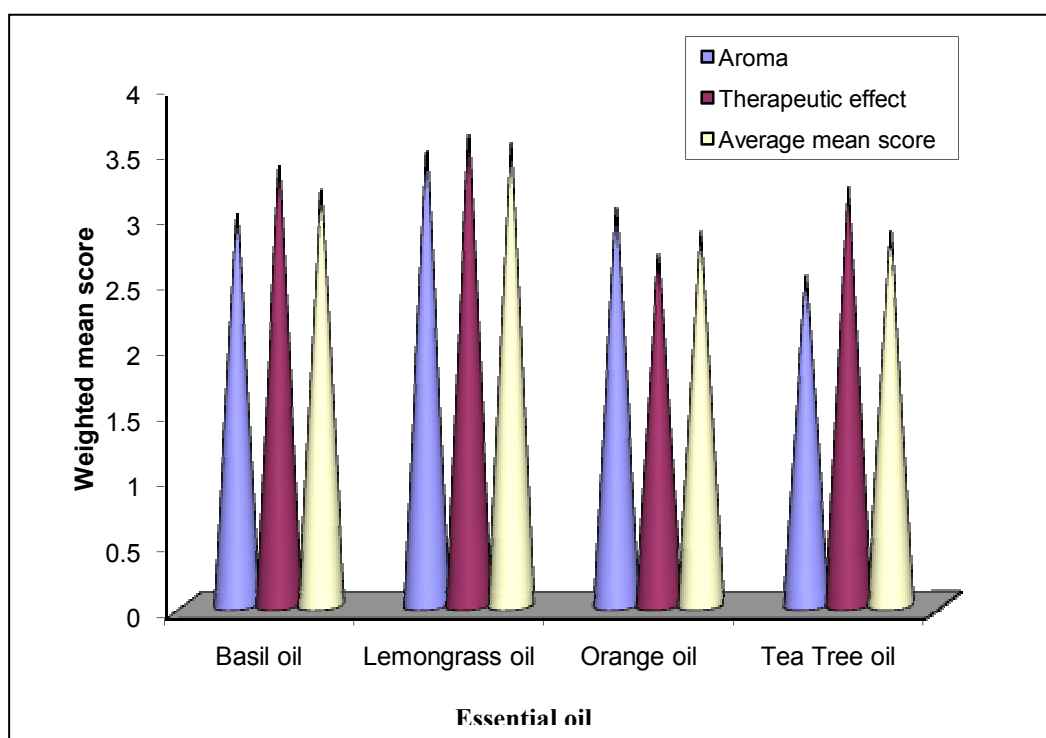
Four aroma oils were got evaluated by 20 respondents on the basis of aroma of the oil and its therapeutic effects using a preferential choice index. The data related to preferences of respondents regarding the essential oils is presented in Table 2.

The data in Table 2 elucidates the average weighted mean score on two parameters i.e. aroma and therapeutic effect. As per ranks given by scientists and students, lemongrass oil scored highest (3.54) ranked Ist followed by basil oil (3.18) ranked IInd and orange oil and tea tree oil (2.86) both ranked IIIrd.

Table 2: Preferences of the respondents for essential oil for microencapsulation

Essential oil	Parameters		Average mean score	Rank
	Aroma	Therapeutic effect		
	WMS			
Basil oil	3.00	3.36	3.18	II
Lemongrass oil	3.48	3.60	3.54	I
Orange oil	3.04	2.68	2.86	III
Tea Tree oil	2.52	3.20	2.86	III

n=20



WMS – Weighted Mean Score

Fig. 4.1 : Preferences of the respondents for essential oil for microencapsulation

It is thus concluded that the most preferred essential oil was lemongrass oil which was used for further experimental work

4.3 Standardization of Microencapsulation Process

Microcapsule gel was prepared using lemongrass oil, gum acacia and gelatin using the complex coacervation technique and optimization of various variables was carried out to obtain the best results.

4.3.1 Optimization of the proportion of lemongrass oil in microcapsule gel:

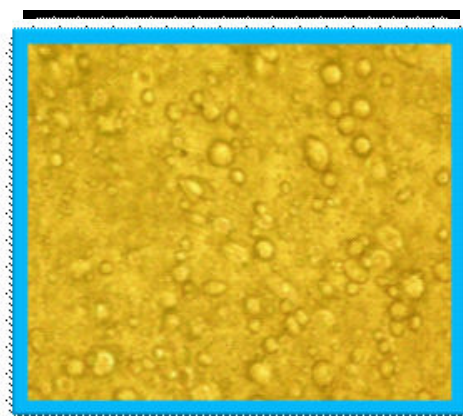
Lemongrass oil forms the core material of the microcapsule and is basically responsible for the fragrance and therapeutic effect. Microcapsule gel was prepared using different ratio of lemongrass oil i.e. 0.5, 1.0, 2.0, 2.5 and 3.0. The gel was observed under inverted microscope

to ensure the presence of microcapsules. Optimum ratio of lemongrass oil was selected on the basis of visual assessment of three parameters i.e. size of microcapsules, uniformity in size and distribution and wall of microcapsules.

The data presented in Table 3 and visual assessment of microcapsule gel indicate that though microcapsules were formed only with one ratio of gum, oil and gelatin i.e. 4:1:4 respectively (Image 1) but were very large in size, had poor uniformity in size and distribution and the wall was very thin. This ratio of lemongrass i.e. 4:1:4 was used for further optimization to achieve the best results.

Table 3: Optimization of ratio of lemongrass oil in microcapsules gel

Ratio of Gum: oil: gelatin	Formation of microcapsules	Parameters		
		Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules
4: 0.5 : 4	No	-	-	-
4 : 1.0 : 4	Yes	Very large	Very poor	Very thin
4 : 1.5 : 4	No	-	-	-
4 : 2.0 : 4	No	-	-	-
4 : 2.5 : 4	No	-	-	-



Microcapsules 4:1:4

Image 1- Microcapsule at optimum ratio of lemongrass oil

4.3.2 Optimizations of ratio of gum acacia in microcapsule gel: Gum acacia forms the wall/ outer core of the microcapsule and protects the oil from abrasion, sunlight and biodegradation thus provides a controlled release to the oil. Microcapsule gel was prepared using different ratio of gum acacia i.e. 1, 2, 3, 4 and 5. The prepared gel was observed under inverted microscope to check the presence of microcapsules. Optimum ratio of gum acacia was selected on the basis of visual assessment of microcapsule gel on three parameters i.e. size of microcapsule, uniformity in size and distribution and wall of microcapsules.

The data presented in Table 4 and visual assessment of microcapsule gel indicates that microcapsules were formed in three ratios of gum, oil and gelatin i.e. 2:1:4, 3:1:4 and

4:1:4 (Image 2). The microcapsules formed in the ratio of 2:1:4 were medium sized, had uniformity in size and distribution and the wall was also sharp and thick as compared to the capsules in the other two ratios i.e. 3:1:4 and 4:1:4, as these were larger in size with average and poor uniformity and thin walls. Therefore the ratio of 2:1:4 was optimized.

Table 4: Optimization of ratio of gum acacia in microcapsule gel

Ratio of gum: oil: gelatin	Formation of microcapsules	Parameters			Rank
		Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	
1:1:4	No	-	-	-	-
2:1:4	Yes	Medium	Average	Sharp and thick	I
3:1:4	Yes	Large	Poor	Thin	III
4:1:4	Yes	Very large	Very poor	Very thin	II
5:1:4	No	-	-	-	-

Oil ratio: 1

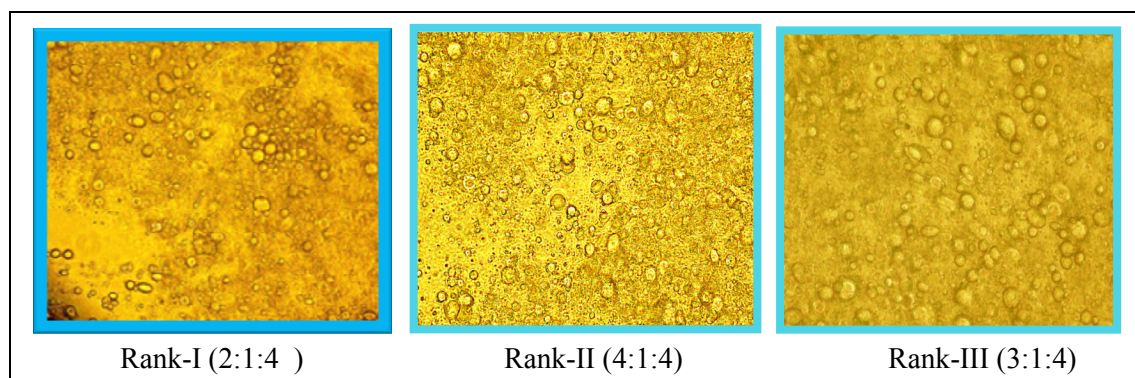


Image – 2: Microcapsules at different ratios of gum acacia

4.3.3 Optimizations of ratio of gelatin in microcapsule gel: Gelatin is a common ingredient of complex coacervation process and gives best results with gum acacia and oil. Microcapsule gel was prepared using different ratio of gelatin i.e. 1, 2, 3, 4 and 5. The gel was observed under inverted microscope to check the presence of microcapsules. Optimum ratio of gelatin was selected on the basis of visual assessment of three parameters i.e. size of microcapsule, uniformity in size and distribution and wall of microcapsules.

The data presented in Table 5 and visual assessment of microcapsule gel indicate that microcapsules were formed in the ratio of gum, oil and gelatin i.e 2:1:3 and 2:1:4 (Image 3). The microcapsules formed in the ratio of 2:1:4 were medium sized, had uniformity in size and distribution and the wall was also sharp and thick as compared to the capsules in the ratio of 2:1:3 which were larger in size with poor uniformity and thin walls. Therefore the ratio of 2:1:4 was optimized for gelatin.

Table 5 Optimization of ratio of gelatin in microcapsule gel

Ratio of gum:oil: gelatin	Formation of microcapsules	Parameters			Rank
		Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	
2:1:1	No	-	-	-	-
2:1:2	No	-	-	-	-
2:1:3	Yes	Large	Poor	Poor	II
2:1:4	Yes	Medium	Average	Sharp and thick	I
2:1:5	No	-	-	-	-

Gum: oil - 2:1

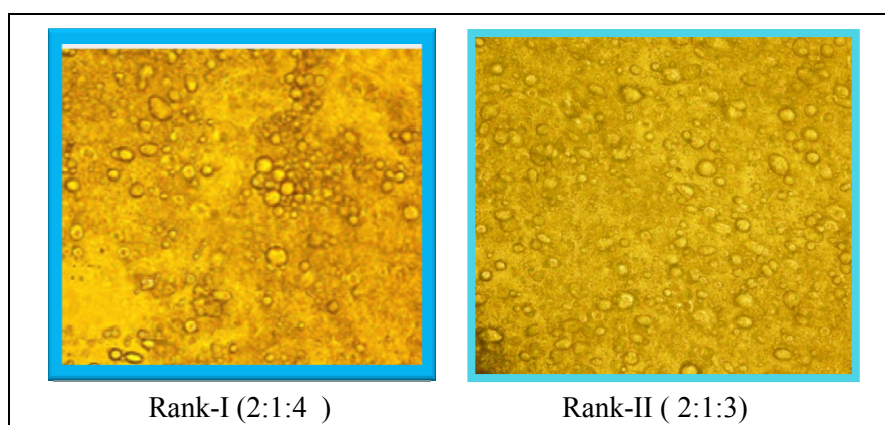


Image-3: Microcapsules at different ratios of gelatin

4.3.4 Optimization of temperature for microencapsulation: For optimization of temperature microencapsulation process was carried out at five different temperatures i.e. 30, 40, 50, 60 and 70°C. The microcapsule gel was observed under inverted microscope to check the presence of microcapsules. Optimum temperature was selected on the basis of visual assessment of microcapsule gel on three parameters i.e. size of microcapsule, uniformity in size and distribution and wall of microcapsules.

It is clear from Table 6 that at 30 and 70°C temperature microcapsules were not formed. The microcapsules formed at 50°C were medium sized, had uniformity in size and distribution and the wall was also sharp and thick as compared to microcapsules formed at 40 and 60°C , as these were medium sized, had average uniformity, sharp and thick walls and large in size, poor uniformity with weak and ruptured walls respectively (Image 4). Hence 50°C temperature was optimized for microencapsulation as at higher temperature the walls get ruptured and size of microcapsule increased.

Table 6: Optimization of temperature for microencapsulation

Temperature (°C)	Formation of microcapsules	Parameters			Rank
		Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	
30	No	-	-	-	-
40	Yes	Medium	Average	Sharp and thick	II
50	Yes	Medium	Good	Sharp and thick	I
60	Yes	Large	Poor	Weak and ruptured	III
70	No	-	-	-	-

Gum:oil:gelatine- 2:1:4

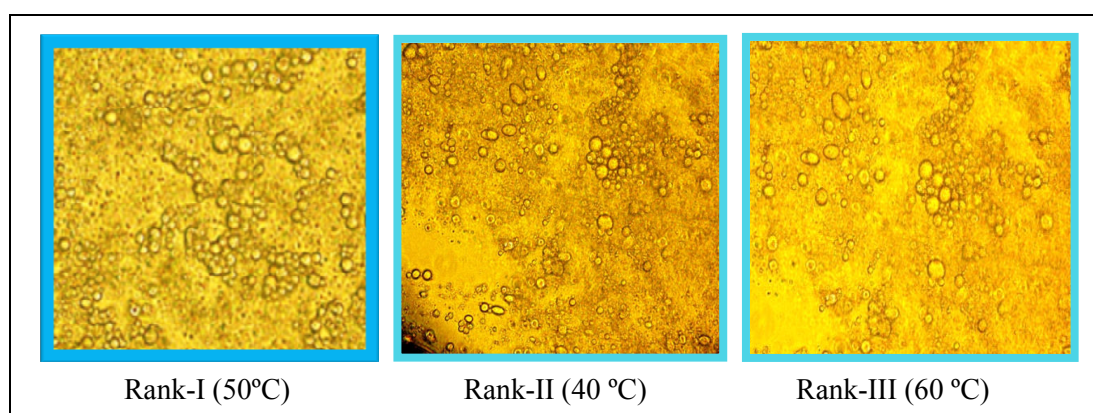


Image-4: Microcapsules at different temperature

4.3.5 Optimization of pH for microencapsulation: The role of pH is very important in microencapsulation as it is responsible for phase separation that leads to capsule formation. To optimize initial pH and the final pH, microcapsule gel was initially set at pH 4.0, 4.5, 5.0, 5.5, 6.0, 6.5 and 7.0 . After the completion of microencapsulation process and gel formation the final pH of the gel was set at 7.0, 7.5, 8.0, 8.5, 9.0, 9.5 and 10. Lower pH was not used as acidic pH might damage the cotton fabric. Microencapsulation was carried out with optimized ratio of gum: oil: gelatin, temperature and pH.

Table 7 : Formation of microcapsules at different pH

Initial pH	4.0	4.5	5.0	5.5	6.0	6.5	7.0
Final pH	Formation of microcapsules						
7.0	x	x	x	x	x	x	x
7.5	x	x	x	x	x	x	x
8.0	x	x	x	x	x	x	
8.5	x	x	x	x	x	x	x
9.0	Formed	Formed	Formed	x	x	x	x
9.5	Formed	Formed	Formed	x	x	x	x
10.0	Formed	Formed	Formed	x	x	x	x

Gum:oil:gelatin-2:1:4, temperature- 50°C

Table 7 unveils that microcapsules were formed only when the initial pH ranged from 4 to 5 and final pH ranged from 9 to 10. The pH range other than this was not suitable for phase separation and microcapsule formation with lemongrass oil.

Table 8 : Optimization of pH for microencapsulation

pH (initial / final)	Formation of microcapsules	Parameters			Rank
		Size of microcapsules	Uniformity in size and distribution	Wall of microcapsules	
4.0/9.0	Yes	Small	Poor	Thin	VII
4.0/9.5	Yes	Small	Average	Thin	IV
4.0/10.0	Yes	Small	Average	Thin	IV
4.5/9.0	Yes	Small	Average	Thick and sharp	II
4.5/9.5	Yes	Medium	Good	Thick and sharp	I
4.5/10.0	Yes	Large	Poor	Thin	VII
5.0/9	Yes	Small	Average	Thin	IV
5.0/9.5	Yes	Medium	Average	Thin	III
5.0/10.0	Yes	Large	Poor	Very thin	IX

It is evident from Table 8 and visual analysis of microcapsule gel that the microcapsules formed at initial pH 4.5 and final pH 9.5 were medium in size with good uniformity in size and distribution with thick and sharp walls (Image -5). Therefore these were selected for further research work as optimum initial and final pH for microencapsulation process.

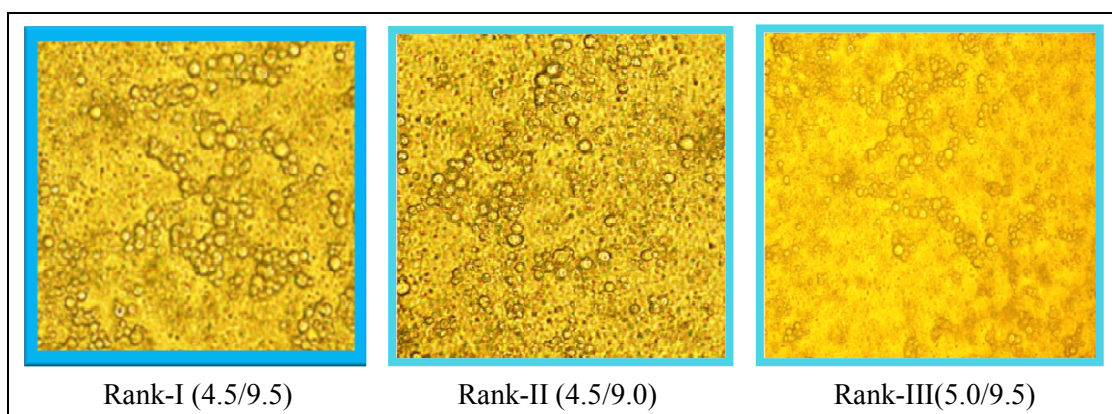


Image-5: Microcapsules at different initial and final pH

Thus it can be concluded that best results were obtained when the ratio of gum: oil: gelatin was 2:1:4, temperature 50°C, initial pH 4.5 and final pH 9.5. The microcapsules formed with these optimized conditions were medium sized, had good uniformity in size and distribution with sharp and thick walls.

4.4 Standardization of Padding Process

For standardization of padding process various variables of padding bath and curing were optimized on the basis of the presence of microcapsules on the fabric examined through e-scope on comparative basis, washing durability, bending length and flexural rigidity.

4.4.1 Optimization of the proportion of microcapsule gel in padding bath: Padding bath was prepared using binder, softener and microcapsule gel. The proportion of binder and softener was kept constant at 10 and 1 respectively and three different ratio of microcapsule gel i.e. 30, 40 and 50 (g/l) was used to prepare the padding bath. The fabric was dipped into the bath with 110-120 expressions and passed through padding mangle and cured at 80-85°C. Optimum ratio of microcapsule gel was selected on the basis of presence of microcapsules on the fabric observed under e-scope, wash durability, bending length and flexural rigidity.

The data presented in Table 9 and visual analysis of treated fabric indicated that at 10:1:50 ratio of binder:softener:microcapsule gel many numbers of microcapsules were present on the fabric surface (Image 6) and their wash durability lasted till 15 wash cycles with an average bending length of 2.47 cm and flexural rigidity of 29.98 mg-cm. Thus the proportion 50 (g/l) was optimized for microcapsule gel and further experiments were carried out.

Table 9: Optimization of the proportion of microcapsule gel in padding bath

Proportion of binder: softener : microcapsules gel	Presence of microcapsules on fabric	Parameters										Rank
		Wash durability (Wash cycles)						Bending length (cm)		Average bending length (cm)	Flexural rigidity (mg-cm)	
		5	10	15	20	25	30	Warp	Weft			
10:1:30	Very few	✓	×	×	×	×	×	2.7	1.83	2.26	25.19	II
10:1:40	Few	✓	✓	×	×	×	×	2.85	1.96	2.40	28.48	III
10:1:50	Many	✓	✓	✓	×	×	×	2.94	2.0	2.47	29.98	I

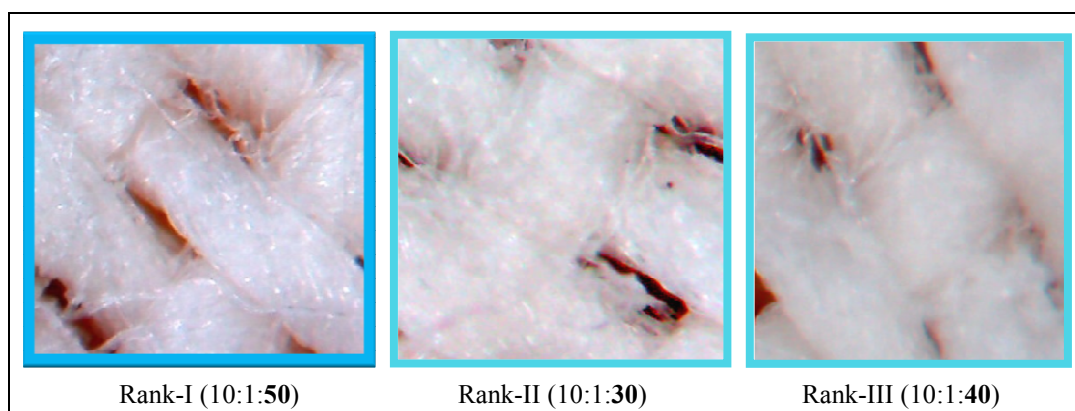


Image-6: Presence of microcapsules on fabric at different proportions of microcapsule gel

4.4.2 Optimization of proportion of softener in padding bath: Different proportions of softener i.e. 1, 3 and 5 were tried with the optimized proportion of microcapsule gel and keeping microcapsule gel and binder proportion constant at 50 (g/l) and 15 respectively. The padding process was carried out and optimization of proportion was done as explained in section (4.4.1)

The data presented in Table 10 and visual analysis of treated fabric indicates that at 10:1:50 ratio of binder: softener: microcapsule gel many numbers of microcapsules were present on the fabric surface (image 7) and their wash durability lasted till 15 wash cycles with an average bending length of 2.43 cm and flexural rigidity of 29.73 mg-cm. It was observed that with further increase in softener ratio lesser number of microcapsules were present on fabric surface and their wash durability also decreased. Thus the proportion 1 was optimized for softener and further optimizations were carried out.

Table 10 : Optimization of proportion of softener in padding bath

Proportion of binder: softener : microcapsules gel	Parameters											Rank
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Bending length (cm)		Average bending length (cm)	Flexural rigidity (mg-cm)	
		5	10	15	20	25	30	Warp	Weft			
10:1:50	Many	✓	✓	✓	×	×	×	2.43	1.99	2.43	29.73	I
10:3:50	Few	✓	×	×	×	×	×	2.87	1.94	2.40	28.39	II
10:5:50	Very few	✓	×	×	×	×	×	2.70	1.90	2.30	26.16	III

Microcapsule gel – 50(g/l)

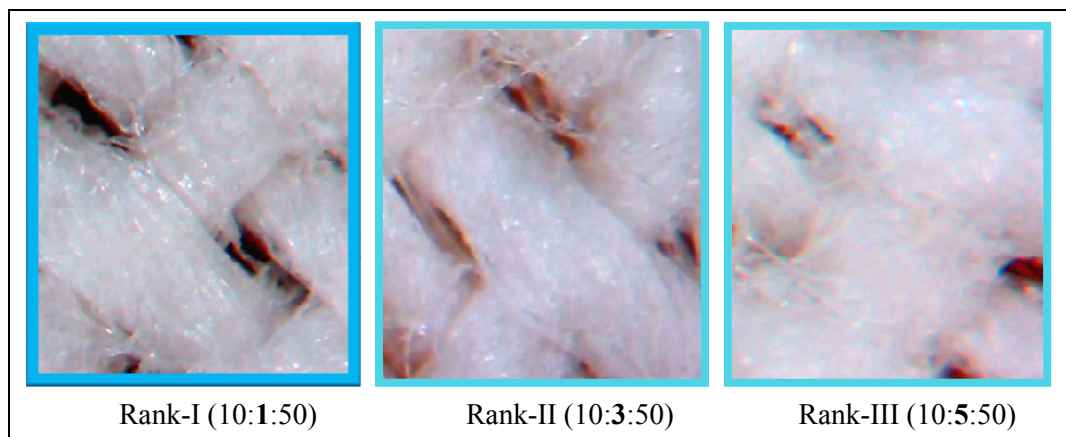


Image-7 : Presence of microcapsules on fabric at different proportions of softener

4.4.3 Optimization of proportion of binder in padding bath: Different proportions of binder i.e. 10, 15 and 20 were tried with the optimized proportion of microcapsule gel and binder. The padding process was carried out and optimization of proportion was done as explained in section (4.4.1)

It is clear from Table 11 and visual analysis of treated fabric that many numbers of microcapsules were present on the fabric surface in all the three proportions i.e. 10:1:50, 15:1:50 and 20:1:50 (Image 9) The wash durability at proportion 10:1:50 lasted till 15 wash cycles whereas for both the other proportions i.e. 15:1:50 and 20:1:50 the wash durability lasted till 20 wash cycles. Hence to optimize the ratio of binder bending length and flexural rigidity were compared and it was observed that fabric treated with 15:1:50 proportion of binder, softener and microcapsule gel had less average bending length (2.40 cm) and flexural rigidity (28.44 mg-cm) as compared to fabric treated with 20:1:50 proportion with an average bending length of 2.49 cm and flexural rigidity of 30.73 mg-cm. . Thus the proportion 15 of binder was optimized and further optimizations were carried out.

Table 11 : Optimization of the proportion of binder in padding bath

Proportion of binder: softener : microcapsules gel	Presence of microcapsules on fabric	Parameters										Rank
		Wash durability (Wash cycles)						Bending length (cm)		Average bending length (cm)	Flexural rigidity (mg-cm)	
		5	10	15	20	25	30	Warp	Weft			
10:1:50	Many	✓	✓	✓	×	×	×	2.93	1.99	2.46	29.73	III
15:1:50	Many	✓	✓	✓	✓	×	×	2.86	1.95	2.40	28.44	I
20:1:50	Many	✓	✓	✓	✓	×	×	2.94	2.05	2.49	30.73	II

Softener: microcapsule gel- 1:50

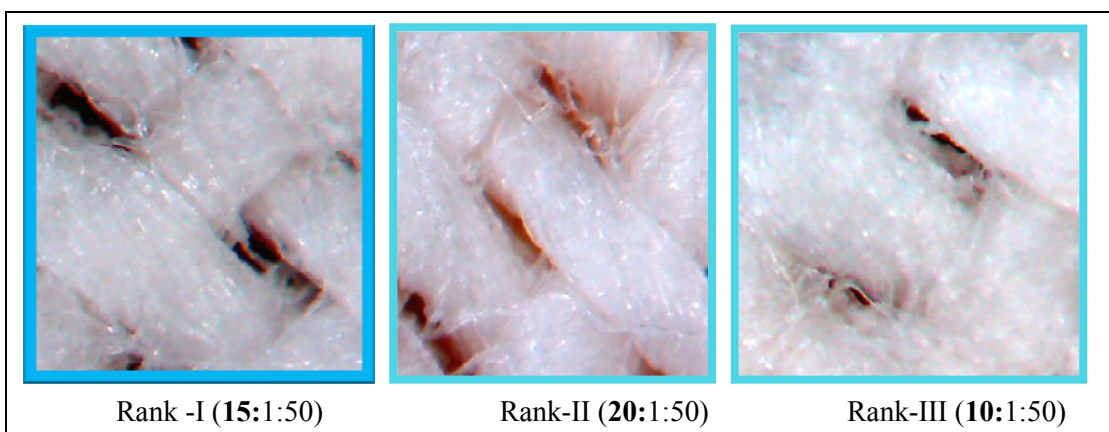


Image-8 : Presence of microcapsules on fabric at different proportions of binder

4.4.4 Optimization of MLR of padding bath : The padding bath was prepared with different MLR i.e. 1:20, 1:30 and 1:40 with the optimized variables. The padding process was carried out and optimization of MLR was done as explained in section (4.4.1)

Data in Table 12 and visual analysis of treated fabric revealed that at MLR 1:20, too many microcapsules were present on the fabric surface and their wash durability lasted till 20 wash cycles with an average bending length and flexural rigidity of 2.45 cm and 29.43 mg-cm respectively. It was observed that with further increase in MLR the presence of microcapsules

on fabric as well the wash durability decreased. Thus the MLR 1:20 was optimized and further optimizations were carried out.

Table 12 Optimization of MLR of padding bath

MLR	Parameters											Rank
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Bending length (cm)		Average bending length	Flexural rigidity (mg-cm)	
		5	10	15	20	25	30	Warp	Weft			
1:20	Too many	✓	✓	✓	✓	×	×	2.90	1.99	2.45	29.43	I
1:30	Many	✓	✓	✓	×	×	×	2.91	1.97	2.44	29.23	II
1:40	Very few	✓	×	×	×	×	×	2.90	1.98	2.44	29.28	III

Binder: softener: microcapsule gel- 15:1:50

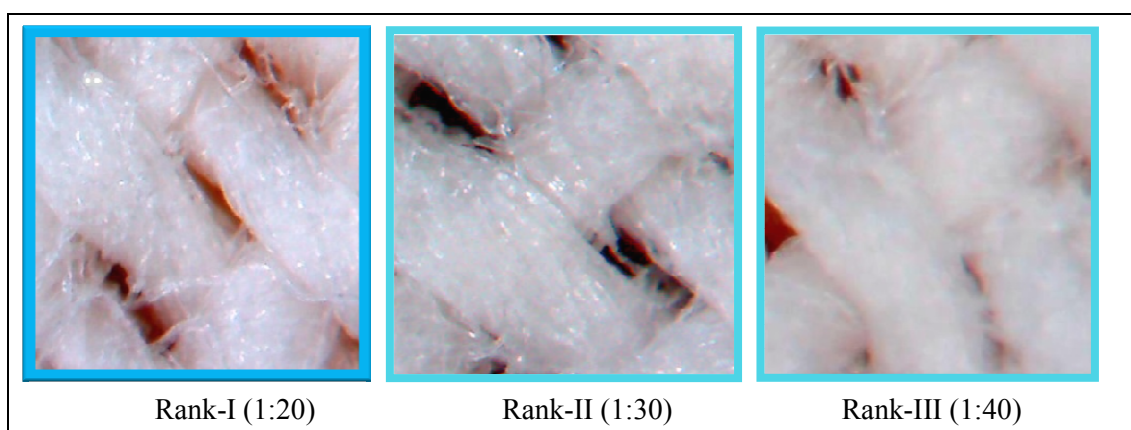


Image-9: Presence of microcapsules on fabric at different MLR

4.4.5 Optimization of curing temperature: The fabric was dipped in the padding bath with optimized proportions of variables and was cured at various temperatures i.e. 70, 80 and 90°C. The padding process was carried out and optimization of temperature was done as explained in section (4.4.1)

The data presented in Table 13 and visual analysis of treated fabric clearly depict that at 80°C temperature too many numbers of microcapsules were present on the fabric (Image 10) and the wash durability lasted till 25 wash cycles with an average bending length and flexural rigidity of 2.46 cm and 29.88 mg-cm respectively. Therefore 80°C temperatures was optimized for curing.

Table 13 : Optimization of curing temperature

Temperature (°C)	Parameters											Rank
	Presence of microcapsules on fabric	Wash durability (wash cycles)						Bending length (cm)		Average bending length (cm)	Flexural rigidity (mg-cm)	
		5	10	15	20	25	30	Warp	Weft			
70	Too many	✓	✓	✓	✓	×	×	2.92	1.98	2.45	29.48	II
80	Too many	✓	✓	✓	✓	✓	×	2.93	2.0	2.46	29.88	I
90	Average	✓	✓	×	×	×	×	2.88	1.97	2.42	28.96	III

Binder: softener: microcapsule gel – 15:1:50, MLR- 1:20

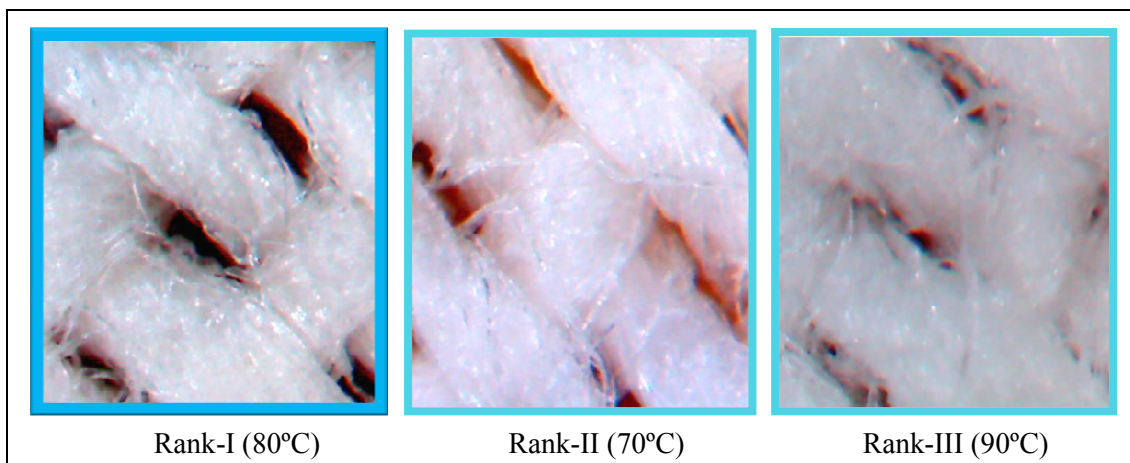


Image-10 : Presence of microcapsules on fabric at different curing temperatures

4.4.5 Optimization of curing time: The fabric padded with the optimized variables of padding bath and curing temperature was cured for different time durations i.e. 30, 60 and 90 sec and optimization was done as explained in section (4.4.1)

It is clear from Table 14 and visual analysis of treated fabric that at curing time of 60 sec too many numbers of microcapsules were present on the fabric (Image 11) with wash durability of 25 wash cycles and average bending length and flexural rigidity of 2.46 cm and 29.98 mg-cm respectively. Therefore 60 sec curing time was optimized for curing process.

Table 14 : Optimization of curing time

Time (sec)	Parameters										Rank	
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Bending length (cm)		Average bending length		Flexural rigidity (mg-cm)
		5	10	15	20	25	30	Warp	Weft			
30	Many	✓	✓	✓	x	x	x	2.90	1.98	2.44	29.28	II
60	Too many	✓	✓	✓	✓	✓	x	2.93	2.0	2.46	29.98	I
90	Many	✓	✓	✓	x	x	x	2.95	2.1	2.52	31.59	III

Binder: softener: microcapsule gel – 15:1:50, MLR- 1:20, temperature- 80°C

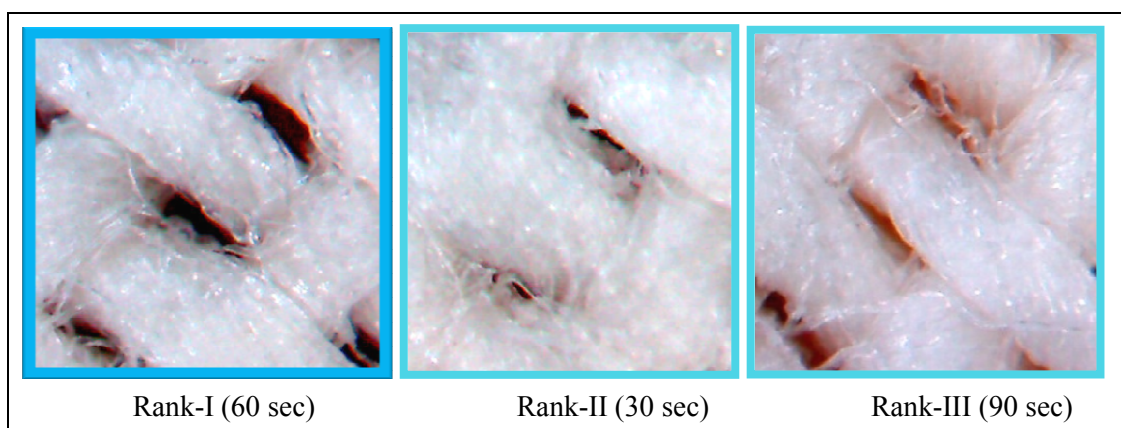


Image-11 : Presence of microcapsules on fabric at different curing time

Thus it can be concluded that best results were obtained when the proportion of binder:softener:microcapsule gel was 15: 1: 50, MLR 1:20, curing temperature 80°C and curing time 60 sec. It was observed that too many microcapsules were present on the fabric surface with these optimized conditions, their wash durability lasted till 25 wash cycles, the average bending length was 2.46 cm and flexural rigidity was 29.98 mg-cm.


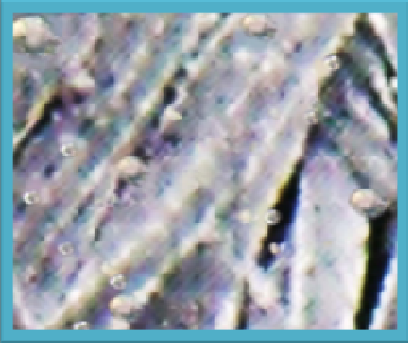
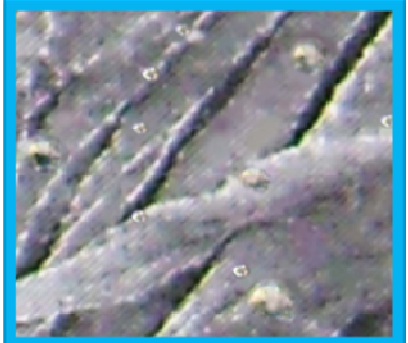

4.5 Assessment of Microencapsulated Fabrics for Wash Durability




The wash durability of microencapsulated fabrics is of considerable importance to the consumers for longer life of the finish. The wash durability does not only depends on the variables of microencapsulation process but also on the padding bath variable ratio, curing temperature and curing time. The presence of microcapsules was examined in treated samples after 5, 10, 15, 20, 25 and 30 wash cycles through microscopic analysis. The retention and intensity of aroma was also checked after wash.

4.5.1 Microscopic analysis of microencapsulated cotton fabric: The presence of microcapsules on treated and washed samples after different number of wash cycles was examined under scanning electron microscope as explained in section (3.8.1)

Table 15 clearly depicts that unwashed samples have too many medium sized microcapsules present on the fabric. After 5 wash cycles some medium and some large microcapsules were present on the fabric, further after 10 washes there was presence of some large and few small microcapsules. Presence of few large microcapsules can be seen on the fabric after 15 wash cycles and presence of very few large microcapsules after 20 washes and 25 wash cycles. After 30 washes there was almost negligible presence of microcapsules. The size and number of microcapsules was judged on comparative basis.

Table 15 : Microscopic analysis of microencapsulated cotton fabric

Wash cycles	Microscopic Analysis	Inference
Unwashed		Presence of too many medium sized microcapsules
5		Presence of some medium and some large microcapsules
10		Presence of some large and few small microcapsules
15		Presence of few large microcapsules

20		Presence of very few large microcapsules
25		Presence of very few large microcapsules
30		Negligible presence

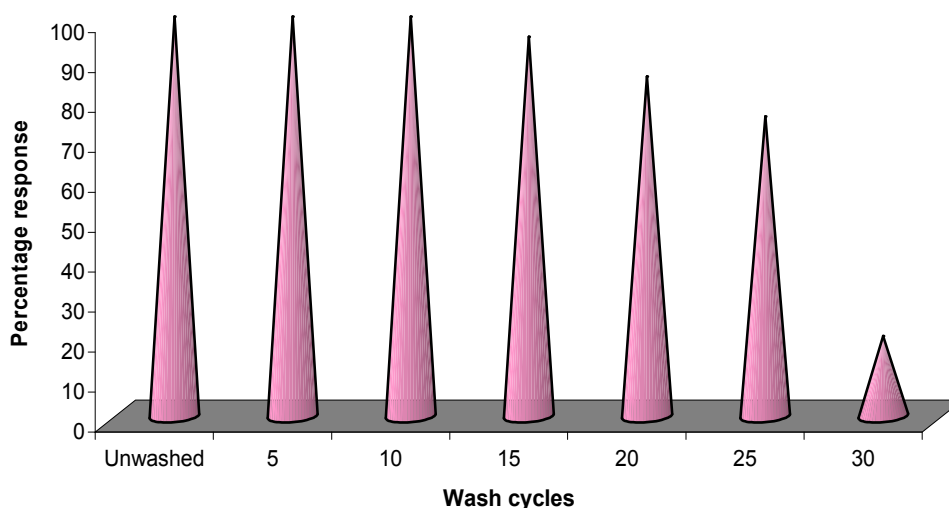
4.5.2 Retention of aroma in microencapsulated fabric after wash : The treated and washed fabric samples were got evaluated on the basis of olfactory analysis by 20 respondents and the responses were taken as ‘yes’ or ‘no’ against aroma retention as explained in section (3.8.2).

The data presented in Table 16 reveals that cent percent respondents agreed that aroma was retained in the treated samples till 10 wash cycles followed by 95, 85, 75 and 20 percent respondents who reported that aroma was retained till 15, 20, 25 and 30 wash cycles respectively. Hence it is concluded that as the number of wash cycles increased the intensity of aroma decreased.

Table 16 : Retention of aroma in microencapsulated fabrics after wash

n=20

Wash cycles	Unwashed	5	10	15	20	25	30
Retention of aroma F (%)	20 (100)	20 (100)	20 (100)	19 (95)	17 (85)	15 (75)	4 (20)

**Fig. 2 : Retention of aroma in microencapsulated fabrics after wash**

4.5.3 Intensity of aroma in microencapsulated fabric after wash

The treated and washed samples were got evaluated against intensity of aroma by 20 respondents on a 5 point scale as explained in section (3.8.3).

Table 17 reveals that the intensity of aroma of unwashed sample (4.35) and after five wash cycles (4.2) was very strong followed by samples after 10 and 15 wash cycle with strong (3.7) and moderate (2.95) aroma respectively. It was observed that as the number of wash cycles increased intensity of aroma decreased. It can be observed that after both 20 (2.5) and 25 (2.3) wash cycles mild aroma was present which further reduced to faint(1.2) after 30 wash cycles

It is thus concluded that intensity of aroma was strong during the first few washes which further decreased gradually and faint aroma was present even after 30 washes.

Table 17 : Intensity of aroma in microencapsulated fabrics after Wash

Wash cycles	Intensity of aroma	Rank
	(WMS)	
Unwashed	4.35	I
5	4.2	II
10	3.7	III
15	2.95	IV
20	2.5	V
25	2.3	VI
30	1.2	VII

4.2 – 5 = Very strong, 3.4 - 4.2= Strong, 2.6 - 3.4 = Moderate, 1.8 - 2.6= Mild and 1 - 1.8 = Faint

4.6 Effect of Finish on Physical Properties of Cotton Fabric

Physical properties of a fabric play an important role for the consumer use. Assessment of physical properties aids in defining the quality of a fabric or those associated with serviceability as there is not always a clear dividing line between physical and performance properties. Thus change in physical properties in terms of fabric count, weight, thickness, bending length, flexural rigidity, tensile strength and elongation of treated and untreated samples was tested using standard test methods.

Table 18: Physical properties of microencapsulated cotton fabric

Sample	Physical Properties										
	Fabric Count		Fabric weight (g/sq.cm)	Fabric thickness (mm)	Bending length (cm)		Flexural rigidity (mg-cm)	Tensile strength (kg)		Elongation (%)	
	Warp	Weft			Warp	Weft		Warp	Weft	Warp	Weft
Controlled	80	57	33.1	27.6	2.1	1.8	14.10	17.0	11.5	19.8	23
Microencapsulated	84	62	51.0	33.5	2.93	2	29.98	13.4	10.2	13.6	17.5
% change	+5	+8.7	+54.1	+21.4	+39.5	+11.1	+112.6	-23.5	-11.3	-16.2	-23.9

+ increase, - decrease

It is clearly observed from the data portrayed in the Table 18 that the controlled fabric had less fabric count (80×57) than the microencapsulated fabric (84×62). Fabric count showed an increase after microencapsulation with a percentage increase of 5 in warp and 8.7 in weft direction. The fabric weight also observed a sharp increase of 54.1 percent after treatment. The fabric weight of controlled sample was 33.1 g/sq.mt which increased to 51.0 g/sq.mt after the application of finish. While examining the fabric thickness, a percentage increase of 24.1 was observed. The controlled fabric had a fabric thickness of 27.6 mm which increased to 33.5 mm after application of finish.

The bending length of the treated samples increased in both warp and weft direction. The bending length of controlled fabric showed a percentage increase of 39.5 in warp

direction and 11.1 in weft direction, a sharp increase of 112.6 percent was also observed in the flexural rigidity of the treated sample.

The data presented in the table shows a percentage loss of 23.5 and 11.3 in tensile strength and 16.2 and 23.9 in elongation in both warp and weft direction respectively.

Thus it can be concluded that there was an increase in fabric count, fabric weight, thickness, bending length of the fabric after microencapsulation thus the flexural rigidity also increased. Tensile strength and elongation decreased but to an acceptable level.

This chapter presents the discussion regarding the findings of the study. The relevant discussion has been presented under the following subheads:

- 5.1 Standardization of microencapsulation process
- 5.2 Standardization of padding process
- 5.3 Assessment of microencapsulated fabrics for wash durability
- 5.4 Effect of finish on physical properties of cotton fabric

5.1 Standardization of Microencapsulation Process

The various variables of the microencapsulation process were optimized to obtain the best results

5.1.1 Optimization of ratio of gum acacia, lemongrass oil and gelatine : Gum acacia and gelatine were used as wall material and lemongrass as core material for the preparation of microcapsules through complex coacervation technique. Various proportions of gum, oil and gelatine were tried individually keeping other variables constant and the gel formed was observed under inverted microscope for the presence of microcapsules.

The ratio of gum acacia:lemongrass oil:gelatin for microencapsulation was 2:1:4 as it formed medium sized microcapsules with uniformity in size and distribution and sharp and thick walls. More amount of oil leads to no gel formation as the gum and gelatine are not able to form wall around it, when the amount of gum acacia was increased lumps started forming due to disproportionate ratio of oil and gum. Zivdar and Najafi(2004) recommended core to wall ratio as 1:1 and 1:2 while gelatine to gum arabic content to be 2:1. Maya *et al.* (2003) did microencapsulation of oil with gelatine and gum acacia with their concentration being 1% and paraffin oil to be 15 ml per 100 ml.

5.1.2 Optimization of the temperature: The optimum temperature for microencapsulation was 50°C. It was observed that the best microcapsules were obtained at this temperature in terms of size, uniformity in distribution and wall. A further increase in temperature resulted in rupturing of the wall and increase in the size of microcapsule as oils are volatile so they start evaporating which leads to the rupturing of wall and seepage of oils. In the study conducted by Teli, *et al.*(2005) the temperature for microencapsulation was reported at 40°C and Maya *et al.* (2003) reported the temperature used for complex coacervation technique at 45°C.

Agrawal and Goel (2010) also reported that higher temperature leads to increase in evaporation of essential oils resulting in rupturing of wall. It was also revealed that smaller microcapsules have longer life as compared to larger microcapsules.

5.1.3 Optimization of pH : The initial optimum pH for microencapsulation was found to be 4.5 and final 9.5. Microcapsules were formed only when the initial pH ranged between 4 to 5 and final 9 to 10 as the phase separation occurs at acidic range and at the end microcapsules needs to be applied on cotton so final pH was kept in an alkaline range.

Karolia and Mandapa (2007) observed that microencapsulation occurred at acidic conditions as it led to phase separation which was an initial and essential step of coacervation. Danfeng *et al.* (2012) also prepared microcapsules by complex coacervation and reported the coacervation pH to be 4 and final to be 9.

5.2 Standardization of Padding Process

The microcapsule gel prepared from the optimized process was applied on the fabric through the padding process using the pneumatic padding mangle. For standardization of padding process the various variables were optimized and after each stage of optimization fabric was observed under e-scope for the presence of microcapsules. Optimization was based on presence of microcapsule on fabric, wash durability, bending length and flexural rigidity.

5.2.1 Optimization of the proportion of microcapsule gel : The optimum proportion of binder, softener and microcapsule gel in padding bath was found to be 15:1:50. It was observed that with lower proportion of microcapsule gel very few microcapsules were deposited in the fabric and poor wash durability was observed because with the increase in the amount of microcapsule gel the number of microcapsules deposited also decreased. Monllor and Sanchez (2009) revealed through SEM and FTIR analysis that when concentration of microcapsules in padding bath increased their number in the fabric also increased. Monllor *et al.* (2010) used 60 g/l of microcapsules for padding along with resin.

5.2.2 Optimization of the proportion of softener: Softener was used to control the stiffness of the microencapsulated fabric. The optimum proportion of binder, softener and microcapsule gel in padding bath was found to be 15:1:50. Higher amounts of softener hindered the deposition of microcapsules on fabric hence decreasing the wash durability. Sathianarayan *et al.* (2009) through the surface morphological data revealed that microencapsulated fabrics become stiff thus some amount of softener must be incorporated during the application of finish (Nelson, 2002a). Sukumar and Laxmikantha (2009) used ratio of binder and softener as 4:1 for padding.

5.2.3 Optimization of the proportion of binder : Binder improves the microcapsule adhesion to fabrics. The optimum proportion of binder, softener and microcapsule gel in padding bath was found to be 15:1:50. Lesser amounts of binder were not able to adhere microcapsules to the fabric and higher amount showed good washing durability as well presence of microcapsule on the fabric. Nelson, (2002a) pointed out the fact that for microcapsules to survive repeated washing and for application to fibres such as cotton, microcapsules should be applied using a binder. Fabien *et al.* (2009) reported that it's not

necessary to have a high concentration of binder and microcapsules to achieve the best wash durability. Proportion of microcapsule and binder should be optimized. To limit the loss of coating material after wash proportion of padding bath materials should be optimized.

5.2.4 Optimization of MLR : Different material to liquor ratio were tried and it was found that maximum number of microcapsules were present on fabric at material to liquor ratio 1:20 and its wash fastness was also good and lasted till 20 wash cycles so it was optimized. An increase in the MLR reduced the deposition of microcapsules as the fabric was just dipped in the padding bath for few minutes so it could not absorb all the contents hence a lower MLR gave better results. Sukumar and Laxmikantha (2009) also used the MLR 1:20 for applying lavender microcapsules on cotton fabric.

5.2.5 Optimization of curing temperature : The optimum curing temperature was 80°C. At higher temperature the wash durability decreased. Shiqui *et al.* (2009) observed that thermal treatment can give contradictory effects on washing durability of fragrance finished fabrics. Higher temperature and longer curing time usually increased fastness of capsules on textiles with decreased aroma inside the capsules because higher the temperature of curing, more the volatilization of fragrance.

Monllor and Sanchez (2009) observed that fragrance loss from microcapsule occurred at temperature between 90°C to 120°C. Microcapsules could not withstand temperature more than 120°C.

5.2.6 Optimization of curing time : The curing time was optimized at 60 seconds as best results were obtained in terms of presence of microcapsules on fabric, wash durability, bending length and flexural rigidity. Treated fabrics if exposed to high temperature for longer curing time resulted in rupturing of wall and decreased in the number of microcapsules. Monllor and Sanchez (2009) revealed that stronger curing conditions increase binder reaction and improved fixation of capsule but fragrance was volatile so it could not withstand strong curing conditions hence they should not be subjected to high temperature and longer curing time.

5.3 Assessment of Microencapsulated Fabrics for Wash Durability

Wash durability is an important factor to be considered for microencapsulated fabrics as finishing with long term fragrance release properties is a desirable commercial goal.

5.3.1 Microscopic analysis : The treated and washed samples after different number of wash cycles were checked for microcapsule presence under scanning electron microscope. It was observed that with the increase in the number of wash cycles the microcapsules were decreasing in number gradually, capsules were visible on the fabric till 25 wash cycles. It was observed that the number of microcapsules on the fabric remained constant for 20-25 wash cycles. Monllor *et al.* (2010) observed that the number of microcapsules that goes out of the

fabric is higher in the first washing. After 10-15 wash cycles the microcapsules leaving the fabric is not so high and it tends to become constant.

5.3.2 Retention of aroma : Aroma was retained in the fabric till 30 wash cycles. This can be attributed to the fact that the wall of the microcapsules provide covering to the oil thus protecting it from outside environment and providing it a controlled release to give it a longer life. Sukumar and Laxmikantha (2009) also revealed that aroma finish retained till 25 wash cycles. Shah and Goel (2010) also pointed that aoma was retained till 30 cycles of wash, rub and abrasion.

5.3.3 Intensity of aroma : It was observed that as the number of wash cycles increased the intensity of aroma decreased as with each wash few microcapsules gets washed away. Wang *et al.* (2005) observed that after washing the fragrance release rates decreased and the results of sensorial evaluation showed that performance of the fabric lasted 25 cycles of wash and the intensity gradually decreased with increase in the number of washes.

5.4 Effect of Finish on Physical Properties of Cotton Fabric

Physical properties predict behaviour of fabric while in use. The changes in physical properties of the fabric after application of finish were tested. The fabric count, fabric weight, thickness , bending length and flexural rigidity of the treated sample increased whereas the tensile strength and elongation decreased. Increase in fabric count is due to the fact that the application of finish brings the warp and weft closer because of shrinkage in fabric. The increase in weight and thickness can be attributed to the fact that microcapsules gets deposited on the fabric. The fabric becomes stiff and the bending length in both warp and weft direction increases, hence the flexural rigidity also increases. Sathianarayan M.P. (2009) revealed that microencapsulated fabrics become stiff and their crease recovery property also decreases. Karolia and Mandapara (2007) reported the increase in bending length in both the directions due to the mechanical deposition of finishing agents in the open interstices of the fabric. Tensile strength may have decreased due to the adverse effect of various chemicals and high temperature used during the microencapsulation process. Sukumar and Laxmikantha (2009) also reported a decrease in the tensile strength of microencapsulated fabric. From the results it was observed that the elongation of finished samples slightly decreased. The elongation may have decreased due to the coating of the finish on the fabric that restricts the yarns to show their natural elasticity.

Microencapsulation is a technique to prepare microcapsules, small particles that contain an active agent or core-material surrounded by coating or shell of limited permeability. The aroma capsule is a kind of container that provides both space for storing a certain amount of aromatic matter and a protective shield from the effects of sunlight, moisture and oxygen. Release of aroma is by diffusion of the fragrance through the capsule wall and rupture of the capsules. Microcapsulation technology can be used to impart durable functional and aesthetic finish to textiles using essential oil as core material.

Essential oils have been used medicinally at different periods in history. Interest in essential oils has revived in recent decades with the popularity of aromatherapy, a branch of alternative medicine that claims that essential oils and other aromatic compounds have curative effects. Studies indicate that aromatherapy is effective for conditions such as anxiety, depression and boosting cellular immune functions. In many of the studies reviewed, scientists are suggesting further research for possible uses of essential oils as an alternative or complement to conventional medical practices

In this study lemongrass microcapsules have been formed and applied to cotton fabric to impart fragrance as well as therapeutic effect. This will not only provide health benefits to the common masses but will also benefit the agriculture sector as it will ensure increased cultivation of aromatic plants and enhanced rate of essential oil extraction. Aroma finish will add value to cotton so the cultivation, production and export of cotton will also boost.

Therefore the present study was conducted with the following objectives:

- To prepare microcapsules using essential oil and to standardise the process of encapsulation.
- To study the wash durability of treated fabrics
- To study the physical properties of treated fabrics.

Methodology

The suitable medium weight white cotton fabric was selected. Lemongrass oil was selected as the core material through a preferential choice index filled by 20 respondents and gum acacia as wall material. Phase separation- complex coacervation technique was selected to prepare the microcapsules. The fabric was desized, scoured and bleached to prepare the fabric for the microcapsule treatment. The process of microcapsulation was standardized for various variables ie. ratio of gum, essential oil and gelatin, temperature, initial and final pH. Variables were optimized on three parameters i.e. size of microcapsules formed, uniformity in

size and distribution and wall of the microcapsule. These parameters were assessed on comparative basis through visual assessment.

The padding process was also standardized for various variables i.e. proportion of binder, softener and microcapsule gel, MLR, curing temperature and curing time. Variables were optimized on the basis of comparative visual assessment of the presence of microcapsules on fabric surface, wash durability, bending length and flexural rigidity.

Through the standardized process of microencapsulation and padding, lemongrass microcapsule gel was prepared and applied on cotton fabric through padding mangle. The wash durability of treated fabrics was studied through microscopic analysis for presence of microcapsules, retention and intensity of aroma after washing.

The physical properties of the finished and controlled fabric were tested and compared.

Major Findings

Standardization of microencapsulation process: The gum, essential oil and gelatin in the ratio 2:1:4 at temperature 50°C with initial pH 4.5 and final pH 9.5 were optimized as the prepared microcapsules were medium sized, had good uniformity in size and distribution and sharp and thick walls.

Standardization of padding process: The ratio of binder, softener and microcapsule i.e. 15:1:50 with MLR 1:20 cured at 80°C for 60 seconds were optimized as the fabric samples padded with this process had too many number of microcapsules, wash durability of 25 wash cycles with average bending length 2.46 cm and flexural rigidity 29.98 mg-cm.

Assessment of treated fabrics for wash durability: The microscopic analysis of unwashed and washed samples showed the presence of microcapsules on the fabric. Maximum number of microcapsules were present in unwashed samples which kept on decreasing with the number of washes. The presence of microcapsules decreased rapidly during the first 15 washes and became constant afterwards as the number of microcapsules that washed out of the fabric was higher in the first few washing and after 15 it becomes constant. Microcapsules were present till 25 wash cycles. Aroma was retained till 30 washes and the intensity of aroma decreased as the number of wash cycles increased.

Effect on physical properties of treated cotton fabric: The fabric count, fabric weight, thickness, bending length and flexural rigidity of the fabric increased whereas the tensile strength and elongation decreased. The fabric count increased due to the shrinkage of the fabric while the deposition of microcapsules on the fabric resulted increase in fabric thickness and weight. The bending length showed an increase in both warp and weft direction due to the deposition of finishing agent in open interstices of the fabric. There was no adverse effect on the physical properties of treated fabric.

Conclusions

Best microcapsules were formed when lemongrass oil was taken in smaller quantities as compared to wall material. High temperature leads to rupturing of wall hence increase the capsule size which results in decreased wash durability. Medium sized microcapsules show best washing durability as compared to larger ones. Microcapsules were formed only when the initial pH was set at acidic pH between 4-5 as phase separation takes place at this pH only. The proportion of binder, softener and microcapsule i.e. 15:1:50 was optimized for padding process, high proportion of microcapsule gel results in maximum deposition of microcapsules on treated fabric whereas higher proportions of softener inhibits the microcapsule deposition. A lower MLR of 1:20 gave best results. Treated fabrics should not be cured at high temperature more than 80°C and should not be exposed to heat for longer curing time. With the optimized variables and process, aroma in microencapsulated fabrics retained till 30 wash cycles and the microscopic analysis showed the presence of microcapsules till 25 washes, the number decreased gradually with the increase in wash cycles and became constant between 20-25 wash cycles. As the number of wash cycles increased the intensity of aroma decreased. The fabric count, weight and thickness of treated fabric increased hence flexural rigidity increased. Microencapsulation did not have any adverse effect on tensile strength and elongation. Bending length of microencapsulated fabric increased but to an acceptable level.

Recommendations

Research based :

- This kind of study will ensure increased cultivation of aromatic plants and will enhance the rate of oil extraction
- Microencapsulation will add value to cotton therefore this will increase its consumption as well as export
- Microencapsulated fabric will not require any stiffening agent.
- Microencapsulated fabrics will act as mood alleviators and will relieve stress.

Review based:

- Microencapsulated fabrics will also provide UV protection as the UPF value will increase because the interspaces between the fabric gets filled with microcapsules.
- Microencapsulated fabrics should be hand washed and dried in shade so the finish will stay longer.

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ANNEXURE - I

1. Preferential Choice Index for Selection of Essential Oil for Microencapsulation

General Information

1. Name of respondent :

2. Education : Graduate
Postgraduate
Ph.D.

3. Designation :

Specefic Information

a. Pleasantness of Smell

Please give your order of preference for the oil on the basis of pleasantness of smell

S.No.	Essential Oil	Pleasantness of smell				
		Most preferred	Preferred	Moderately preferred	Less preferred	Not preferred
1.	Basil oil					
2.	Lemon Grass oil					
3.	Orange Blossom oil					
4.	Tea-Tree oil					

b. On the basis of therapeutic effect

Please give your order of preference for the oil on the basis of therapeutic effect

S.NO.	Essential oil	Therapeutic effect	Preference				
			Most preferred	Preferred	Moderately preferred	Less preferred	Not preferred
1.	Basil Oil	Cooling up of skin, relieves mental fatigue, improves memory					
2.	Lemon Grass Oil	Stomach aches, diahorrea, depression, insomnia, flatulence, sciatica					
3.	Orange Blossom Oil	Sensitive skin, diahorrea, shock, fear and depression					
4.	Tea-Tree Oil	Antiseptic, germicide, antiviral, relieves pain, cold and cough					

ANNEXURE - II

Evaluation Performa for Intensity of Aroma in Microencapsulated Fabrics after Wash

General Information

1. Name of respondent :
2. Education : Graduate
Postgraduate
Ph.D
3. Designation :

a. Rank order on the basis of intensity of smell

Please give the rank order on the basis of intensity of smell

5-very strong 4- strong 3- moderate 2- mild 1- faint

S. No	Sample	Rank
1	A	
2	B	
3	C	
4	D	
5	E	
6	F	
7	G	

ANNEXURE - III

Evaluation Performa for Retention of Aroma in Microencapsulated Fabrics after Wash

General Information

1. Name of respondent :
2. Education : Graduate
Postgraduate
Ph.D
3. Designation :

a. Responses for retention of aroma.

Please give response for the retention of aroma.

S. No	Sample	Yes	No
1	A		
2	B		
3	C		
4	D		
5	E		
6	F		
7	G		

ANNEXURE - IV

Lemon grass microencapsulated cotton fabric

ABSTRACT

Title of thesis : **Microencapsulation of Essential Oil on Cotton**
Full name of the degree holder : Latika Bhatt
Admission No. : 2010HS169M
Title of degree : Masters of Science in Textile and Apparel Designing
Name and address of Major Advisor : Dr. Saroj S. Jeet Singh
Dean, I.C. College of Home Science and
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Degree awarding university : CCS Haryana Agricultural University, Hisar (Haryana)
Year of award of degree : 2012
Major subject : Textile and Apparel Designing
Total no. of pages in thesis : 48 + iii + III
Number of words in abstract : 306

Key words: Microencapsulation, aromatherapy, essential oil

The study was conducted to standardize the process of microencapsulation and padding, to impart aesthetic and functional finish on cotton fabric. Microencapsulation is a technique to prepare microcapsules, small particles that contain an active agent or core- material surrounded by coating or shell of limited permeability and can be used to impart durable functional and aesthetic finish to textiles using essential oil as core material. Lemongrass oil was selected as the core material and complex coacervation technique was used for encapsulation. Ratios of gum, oil and gelatin, temperature and pH were standardized for the process of microencapsulation. The proportion of ingredients for padding bath i.e. microcapsule gel, binder and softener were also standardized along with MLR, curing time and temperature. Cotton fabric was padded with microcapsule gel using the optimized process and the washing durability and physical properties were tested. It was summarized that 2:1:4 ratio of gum, oil and gelatin , at a temperature of 50 with initial and final pH 4.5 and 9.5 respectively was optimized for microencapsulation process. The proportion of binder, softener and microcapsule gel was optimized at 15:1:50 with MLR 1:20 cured at at 80°C for 60 seconds. The samples retained aroma till 30 wash cycles and as the number of wash cycles increased the intensity of aroma decreased. The fabric count, fabric weight and fabric thickness of treated fabric increased hence flexural rigidity increased. Microencapsulation did not have any adverse effect on tensile strength and elongation.. Bending length of microencapsulated fabric increased but to an acceptable level. Microencapsulated fabrics will not only provide health benefits to the common masses but will also benefit the agriculture sector as it will ensure increased cultivation of aromatic plants and enhanced rate of essential oil extraction. Aroma finish will add value to cotton so the cultivation, production and export of cotton will also boost.

MAJOR ADVISOR

DEGREE HOLDER

HEAD OF THE DEPARTMENT

CURRICULUM VITAE

- a) Name of the Student : Latika Bhatt
- b) Date of Birth : 15 June, 1988
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- d) Mother's Name : Mrs. Manorama Bhatt
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- j) Academic Qualifications:



Degree	University/Board	Year of passing	Percentage	Subjects
Secondary Examination	CBSE	2003	84.7	Hindi, English, Maths, Science, Social Science
Senior Secondary Examination	CBSE	2005	67.4	Hindi, English, Maths, Physics, Chemistry
B.Sc. (Home Science)	GBPUA&T, Pantnagar	2010	7.89	F&N, HDFS, CT, FRM, Extension
M.Sc. (Home Science)	CCSHAU, Hisar	2012	8.71 (Pursuing)	TAD

- k) Co-curricular activities :
- Attended RAWE training for 2 months.
 - Acquired 2 months implant training from Shruti Foundation, a domestic manufacturing unit at Noida
 - NSS 'B' and 'C' certificate holder.
 - Held the position of Vice President of SPIC MACAY, Pantnagar Chapter.
 - Organized and participated in various university level events.
 - Won science quiz competition at district level (Nainital).
 - Won various prizes in debates, essay writing, rangoli competitions at school , college & university level.
 - Attended National seminar on Recent Innovation in Engineering and Technology, Ambala , Micro Finance: Issues and Challenges for Sustainability, Udaipur, Scenario of Women in Agriculture and Future Projections, Hisar.

- l) Medals/Honours received :
- ICAR-JRF fellowship during M.Sc. degree programme.

- m) List of publications :

Paper abstracted and presented:

- Bhatt, L., Singh, S.S.J. and Rose, N.M. 2012. Microencapsulation and Aromatherapeutic Textiles, 3rd National Conference, Recent Innovation in Engineering & Technology (RIET-2012). March-2012. Global Galaxy Group of Institute. pp-47
- Bhatt, L. and Singh, S.S.J. 2012. Microfinance: A Key to Development, National Seminar on Micro Finance: Issues and Challenges for Sustainability. February 23- 24. Udaipur. pp-13

Full length paper:

- Bhatt, L. and Saroj, S.S.J. 2012. Microfinance in handicrafts for Empowering Women. February 16-17. Proceedings of National Seminar on Scenario of Women in Agriculture and Future Projections. Hisar.