

## Original Research Article

### Utilization of Microencapsulated Thyme Essential Oil for Aroma Treatment of Wool Fabric

#### ABSTRACT

**Background:** With rising global trends and changing lifestyles in ~~terms of~~ fashion, beauty as well as healthcare, the awareness of consumers has enforced the evolution of specialty ~~value added~~value-added textiles. Essential oils ~~and basic aromatherapy ingredients which are basic ingredients of aromatherapy~~ are microencapsulated and applied ~~on to the textiles materials~~ to provide a therapeutic effect and ~~long-lasting~~long-lasting aromas. The present study was carried out to prepare thyme essential oil microcapsules using a complex coacervation technique. The prepared microcapsules were applied on wool fabric using pad-dry-cure method by optimizing various variables of aroma treatment.

**Methods:** Thyme essential oil was used ~~on the basis of~~based on its aromatic and therapeutic properties. ~~Complex-A complex~~ coacervation technique of microencapsulation was used ~~for preparation of to prepare~~ thyme oil microcapsules. The padding bath components and treatment variables were optimized ~~on the basis of based on presence of microcapsules on wool fabric as observed under stereo zoom microscope, aroma durability to washing and improvement in the presence of microcapsules on wool fabric as observed under a stereo zoom microscope, aroma durability to washing and improved~~ properties of treated ~~fabric material in terms of regarding~~ bending length, flexural rigidity and crease recovery angle. The aroma treatment was given on wool fabric using pad-dry-cure method.

**Result:** Optimized variables for aroma treatment were 60 g/l microcapsule gel, ~~2-2wog/l softener softeners~~ and ~~10-10g/l binder concentration~~, 1:20 material to liquor ratio, **35 °C** temperature, and 30 minutes treatment time as at these conditions more number of microcapsules, longer wash durability and better fabric properties in terms of bending length, flexural rigidity and crease recovery angle were observed. ~~Aroma treated~~Aroma-treated wool fabric were dried at **70 °C** temperature for 4 minutes and cured at **100 °C** temperature for 60 seconds.

**Conclusions:** ~~The~~ thyme essential oil ~~have~~has many reported therapeutic properties therefore microencapsulated thyme ~~essential basic oil treated oil treated~~ wool fabric can be used for apparel, home, and healthcare textiles. Higher ~~concentration~~concentrations of microcapsule gel and lower concentrations of softener and binder promoted the deposition of a maximum number of

microcapsules on wool fabric. Complex coacervation ~~technique-techniques~~of microencapsulation for preparation of thyme essential oil microcapsules and pad-dry-cure method to impart durable aroma treatment on wool are needed to acquire long term sustained finish.

~~Key words~~**Keywords:** Thyme essential oil microcapsules, wool fabric, optimization, pad-dry-cure method, aroma treatment

## INTRODUCTION

A ~~consumer-oriented~~consumer-oriented21<sup>st</sup>-century21st century challenges garment and fabric manufacturers to ~~come up with revolutions which result from the technological improvement, not only to help in strengthening the existing product but also to develop revolutions resulting from technological improvement, not only to help strengthen the existing product and~~ diversify and flourish in new areas. New textile technologies have empowered the application of ~~new-freshingredient on the fabric to provide its functional benefits to the end product (West and Annett Hitchcock, 2018 and Eyupogluingredients on the fabric to provide functional benefits to the end product (West and Annett-Hitchcock, 2018; Eyupoglu et al., 2021). Functional-Available~~ finishes from the natural substrates ~~comprises of those substances that are obtained from plants and animals possess many advantages such as non-toxic, non-irritant, biodegradable, cost effective, easy availability~~comprise those ~~substances obtained from plants and animals that possess many advantages, such as non-toxic, non-irritant, biodegradable, cost-effective, easy availability,~~ etc. Natural oils such as essential oils are being promoted ~~to be used~~ for finishing application due to their ~~good~~ efficacy without ~~any~~ harmful effects (Naikwadi *et al.*, 2017;~~and~~ Sayed *et al.*, 2022).

Fragrances in ~~form of~~ essential oils and aromatic compounds, when applied ~~on to~~ textile materials, give the textile a pleasant ~~odour-odor~~that ~~gives the wearer~~provides the wearer with maximum beneficial effects. This process is known as aroma finish. The fragrance applied by ~~the~~ use of essential oil not only provides a pleasant smell ~~but also~~and the beneficial effect of aromatherapy. Aromatherapy does not cure conditions but ~~help~~helps the body to find a natural way to ~~cure~~heal itself and improves immune response (Kumar *et al.*, 2021~~and~~; Sousa *et al.*, 2022). Aromatic oily liquids,~~called~~ essential or volatile oils, are obtained from plant materials. Essential oils extracted from different parts of the same plant may have completely different scents and properties. ~~There are various essential oils used in aromatherapy.~~Various essential oils are used in aromatherapy for moisturizing, refreshing, and other wellness effects(Ahmad *et al.*, 2018~~and~~; Hamid *et al.*, 2023).

The thyme essential oil is a combination of ~~monoterpenes~~. The ~~main-primary~~substance of this oil ~~are~~is phenol isomer carvacrol, and ~~it have~~has active biological ~~action-actions~~such as antifungal, antibacterial, antioxidant activity, ~~antitabagism-antinatalism~~, and antispasmodic. Thyme is commonly used as a culinary herb and for different medicinal purposes. Nowadays, thyme ~~present a wide range~~

of functional possibilities in pharmacy, food and cosmetic industry presents various functional possibilities in the pharmacy, food, and cosmetic industries. The interest in the formulation of formulating pharmaceuticals, nutraceuticals, and cosmeceuticals based on thyme is due to the treatment of treating disorders affecting the respiratory, nervous, and cardiovascular systems. Thyme essential oil-treated oil-treated fabric creates the a microclimate for the wearer and control. It control the release of essential oil through friction and movement of body body movement, that which will play a significant role in the health and wellness of the wearer (Salehi *et al.*, 2019 and; Silva *et al.*, 2021).

Textile materials are treated with pleasant odour producing odor-producing essential oils and aromatic compounds to impart an aroma finish so that the wearer gets some valuable effects. Due to their highly volatile nature, these are ineffective to utilize for profitable applications in textile for profitable textile applications. But microencapsulation technology locks essential oils with fibre fiber in a stable manner. Microencapsulation is a process by which individual particles of an active agent can be stored within a shell, surrounded or coated with a continuous film of polymeric material to produce particles in the micrometre-micrometer to millimetre-millimeter range, for protection and/or later release. The unique advantage of microencapsulation lies in that the core material is completely coated and isolated from is that the core material is completely coated and isolated from the external environment. Microencapsulation would not affect the properties of core materials, provided that proper shell material and preparing preparation method methods are chosen (Zhao *et al.*, 2019 and; Rukhaya *et al.*, 2021).

As close friends of human humans, textiles can make aromatherapy easy wherever these are needed. Among all the natural origin fibres fibers, wool plays a significant role in the textile industry. Wool is a natural crude protein fibre fiber and the wool polymer is a linear keratin polymer. Its repeating unit is sulphur containing sulfur-containing amino acid linked together with disulphide bonds (Srivastava and Srivastava, 2017 and Rukhaya acids linked with disulfide bonds (Srivastava and Srivastava, 2017; Rukhaya *et al.*, 2022)). Wool is a very absorbent fibre fiber as it contains a higher amorphous areas, i.e., about 75-70 percent amorphous and 25-30 percent crystalline, however; however, the scaly structure of wool makes it partially water repellent, but when moisture or other substances like oil and aroma once penetrate the fibre fiber surface, get absorbed quickly and has good retention for a longer time.

## MATERIALS AND METHODS

### Materials

Pure griegre woven wool fabric having 59.40 ends/in (EPI), 49.00 picks/inch (PPI), 161 g/m<sup>2</sup> basis weight, and 0.350 mm thickness was procured from the market of Ludhiana city of Punjab, India. Thyme essential oil was purchased from Emmbros Overseas Lifestyle Pvt. Ltd., Haryana, India. Wall materials, i.e., gum acacia and gelatin, softener (silicon), and binder (beta-

cyclodextrin) were provided by chemical suppliers of in Haryana, India. Other materials, such as acetic acid, formalin, sodium hydroxide, and a wetting agent (Ultravon JU), were also used in the study.

### Preparation of Fabric

The wool fabric was weighed initially, and pre-wetting was done for 10-15 minutes. The scouring solution of 2 g/l neutral soap was prepared, maintaining the ~~material-to-liquor~~ material-to-liquor ratio 1:20 and pH 7. The fabric was added to the scouring bath ~~and temperature of bath,~~ and the ~~temperature~~ was raised gradually and ~~maintained-held~~ at 60 °C. The wool fabric was treated for 60 minutes with occasional stirring. The scoured fabric was then rinsed with plain water and dried at room temperature.

### Preparation of Thyme Oil Microcapsules

~~Phase separation complex coacervation technique of microencapsulation was used for preparation of~~ The phase separation-complex coacervation technique of microencapsulation was used to prepare thyme essential oil microcapsules. ~~For standardization of microencapsulation process, the ratios of essential oil, gum and gelatin, temperature and pH were optimized~~ The critical oil, gum, gelatin, temperature, and pH ratios were optimized to standardize the microencapsulation process. The formed microcapsules were examined under an inverted microscope, and ~~on the basis of~~ based on the size, distribution, and quality of the wall of capsules; ~~different; different~~ process variables were optimized; 16 g of gelatin was ~~weighed-weighed~~ and dissolved in 25 ml warm water and stirred using a ~~high-speed~~ high-speed stirrer for 10 minutes; 4 g of vetiver oil was added to the solution at 45 °C; 16 g of gum acacia was weighed and dissolved in 25 ml warm water separately. The gum acacia solution was added to the gelatin solution, and the temperature of the solution was maintained at 45 °C. The pH of the solution was decreased to 4.5 by adding dilute acetic acid and ~~stirred-stirring~~ at high speed for 20 minutes. The pH of the solution was increased to 8.5 using sodium hydroxide solution to form a microcapsule gel. For stabilization, 1 ml of 17 percent alcoholic formalin was added to the formed capsules.

### Standardization of Aroma Treatment for Wool Fabric

For aroma treatment, ~~thyme essential oil microcapsules were applied on wool fabric using pad-dry-cure method~~ the pad-dry-cure method applied thyme essential oil microcapsules on wool fabric. The padding bath components (microcapsule gel, softener, and binder) and other treatment variables, i.e., ~~material-to-liquor~~ material-to-liquor ratio, treatment temperature and time, drying temperature and time, curing temperature and time, were optimized ~~on the basis of~~ based on the presence of microcapsules on wool fabric as observed under stereo zoom microscope, aroma durability to washing and improvement in properties of treated fabric in terms of bending length, flexural rigidity ~~and-and~~ increase recovery angle. The aroma treatment was given to wool fabric using

optimized concentrations and conditions of ~~the pad-dry-cure method of finish application~~ finish application method.

### Optimization of padding bath components

The padding bath was prepared using microcapsule gel, softener and binder. The concentration of padding bath components ~~were was~~ optimized ~~on the basis of~~ based on presence of microcapsules on the fabric as analyzed under a stereo zoom microscope.

**i. Optimization of microcapsule gel concentration:** ~~For determination of~~ To determine the optimum concentration of microcapsule gel, four padding ~~bath baths~~ of different concentrations of microcapsule gel, i.e., 30, 40, 50, and 60 g/l, were prepared. For giving aroma treatment to wool fabric through ~~the~~ pad-dry-cure method, the samples were immersed in the solutions of four different concentrations of microcapsule gel with 5 g/l binder and 1 g/l softener at MLR 1:20, maintaining a temperature of 35 °C for 30 minutes with occasional stirring. ~~Afterwards~~ Afterward, the fabric was placed on the trough of the padding mangle with ~~padding padding~~ solution and passed through the squeezing rollers of the padding mangle at ~~pneumatic pneumatic~~ pressure of 2 kg/cm<sup>2</sup> with two dips and nips having 80-90 percent expression. As the fabric left the padding mangle, it was ~~subsequently~~ dried at 80 °C for 5 minutes and ~~cured cured~~ at 110 °C for 1 minute (Thilagavathi *et al.*, 2007; ~~and~~ Kumari, 2015).

**ii. Optimization of softener concentration:** Four different concentrations of softener, i.e., 1, 2, 3, and 4 g/l were taken to optimize the ~~concentration of softeners~~ softener concentration with optimized concentration of microcapsule gel keeping all other variables constant. Padding, drying and curing ~~was were~~ carried out, and optimized ~~concentration of softeners~~ softener concentration was selected.

**iii. Optimization of binder concentration:** For optimization of ~~concentration of binder~~ binder concentration, four different concentrations of binder, i.e., 5, 10, 15, and 20 g/l, were taken with optimized concentrations of microcapsule gel and softener, while all other variables were kept constant. Padding, drying, and curing ~~was carried out and optimization of binder concentration were~~ carried out, and binder concentration optimization was done.

### Optimization of material-to-liquor ratio

To determine ~~the~~ optimum ~~material-to-liquor~~ material-to-liquor ratio (MLR) of ~~the~~ padding bath, four different ~~material-to-liquor~~ material-to-liquor ratios, i.e., 1:20, 1:30, 1:40, and 1:50, were taken using optimum concentrations of microcapsule gel, softener, and binder ~~while~~. ~~At the same time,~~ other variables of ~~the~~ pad-dry-cure method were kept constant. The optimum M:L ratio was ~~selected on the basis of presence of microcapsules, wash durability and improvement in properties of treated fabric, i.e., fabric~~ selected for the presence of microcapsules, wash durability, and improved properties of treated fabric, i.e., fabric i.e. bending length, flexural rigidity, and crease recovery angle.

### Optimization of treatment temperature

The aroma treatment was given to wool fabric at four different temperatures, i.e., 25, 35, 45, and 55 °C with optimized concentrations (microcapsule gel, softener, and binder) and conditions (M:L ratio) while other variables were kept constant. The ~~padding, drying and curing of the fabric~~ fabric's

~~padding, drying and curing was were~~ carried out, and the temperature exhibiting the best results was selected as the optimum treatment temperature.

### **Optimization of treatment time**

The treatments were carried out for four different time durations ~~i.e. 20, 30, 40 and 50 minutes using optimized concentrations of microcapsule gel, softener and binder, i.e., 20, 30, 40, and 50 minutes using optimized microcapsule gel, softener, and binder concentrations,~~ keeping an optimized M:L ratio and treatment temperature. ~~On the basis of~~ Based on presence of microcapsules, wash durability and improvement in the presence of microcapsules, wash durability, and improved fabric properties in terms of bending length, flexural rigidity, and crease recovery angle, treatment time was optimized for aroma treatment of wool fabric.

### **Optimization of drying temperature**

Drying of treated fabric samples was carried out at four different temperatures, i.e., 60, 70, 80, and **90 °C** for 5 minutes, and subsequently cured at **110 °C** for 1 minute. The drying temperature giving the best results was selected as the optimum drying temperature.

### **Optimization of drying time**

To determine optimum drying time, fabric samples were treated using optimum concentrations of padding bath components, M:L ratio, treatment temperature, and treatment time. The drying of treated samples was carried out for four different time durations, i.e., 2, 3, 4, and 5 minutes at optimum drying temperature, keeping curing temperature and time constant, and optimization of drying time was done.

### **Optimization of curing temperature**

Drying of treated samples was carried out at optimum drying temperature and time, and curing treatment was carried out at four different temperature ranges, i.e. 100, 110, 120, and **130 °C**, keeping ~~curing-fixing~~ time constant and optimization of curing temperature was done.

### **Optimization of curing time**

After ~~application of aroma treatment, the padded samples were dried at optimum temperature and time and cured at optimum temperature for four different durations of applying aroma treatment, the padded samples were dried at optimum temperature and time and cured at optimum temperature for four different~~ curing ~~time-times,~~ i.e., 30, 60, 90, and 120 seconds. ~~On the basis of~~ Curing time was optimized based on the presence of microcapsules, wash durability, and improved properties of aroma-treated fabric in terms of bending length, flexural rigidity, and crease recovery angle ~~presence of microcapsules, wash durability and improved properties of aroma treated fabric in terms of bending length, flexural rigidity and crease recovery angle, curing time was optimized.~~

## **RESULTS AND DISCUSSION**

### **Optimization of Padding Bath Components**

For aroma treatment of wool fabric, a padding bath was prepared using microcapsule gel of thyme essential oil, softener, and binder. The concentrations of padding bath components, i.e., microcapsule gel, softener, and binder, were optimized ~~on the basis of~~ based on the presence of microcapsules on the treated wool fabric, good aroma retention to washing, and ~~improvement in~~ improved properties of the treated fabric.

### Optimization of microcapsule gel concentration

The data in ~~the~~ Table 1 and microscopic assessment of the ~~aroma-treated~~ aroma-treated fabric (Image 1) indicate that when 50 and 60 g/l concentrations of microcapsule gel were used, many microcapsules were present on the surface of the fabric, and their wash durability lasted till 20 wash cycles. At 50 g/l concentration of microcapsule gel, bending length (3.25 cm) and flexural rigidity (15.29 mg\*cm) were observed more ~~and~~. The degree of crease recovery angle was less (114.00°) as analyzed and compared with 60 g/l concentration of microcapsule gel which had decreased bending length (3.22 cm) and flexural rigidity (14.63 mg\*cm) and increased crease recovery angle (114.99°). At other concentrations, ~~presence of few to average number of the presence of few to average~~ microcapsules was seen on the fabric surface with low aroma retention. It is ~~obvious-evident~~ from the table that more ~~number of~~ microcapsules were present at 60 g/l concentration of microcapsule gel with good wash durability, improvement in softness, and good resistance to creasing. Therefore 60 g/l concentration of microcapsule gel was chosen as the optimum concentration for ~~preparing~~ preparation of the padding bath. ~~Thite and Gudiyawar, 2020~~ also used the ratio 50:50 of microcapsule gel and water ~~for preparing the padding bath for application of to prepare the padding bath for applying~~ tulsii, lemongrass, and citronella essential oil on woven cotton fabric. Similar results were reported by ~~Rana et al., 2017~~ and ~~Lim and Sethayanond, 2019~~.

### Optimization of softener concentration:

It is ~~obvious~~ is evident from the Table 2 and microscopic evaluation (Image 2) of the thyme essential ~~oil-treated~~ oil-treated wool fabric that 2 and 3 g/l concentrations of microcapsule gel showed the presence of many microcapsules on the fabric surface with wash durability of aroma treatment lasted till 20 wash cycles. It was found that 2 g/l concentration exhibited ~~improvement in~~ improved softness as indicated by decreased average bending length (3.21 cm) and flexural rigidity (14.79 mg\*cm). Also, ~~an~~ increased crease recovery angle ~~of~~ 116.83° was recorded at 2 g/l concentration of softener as compared to 3 g/l concentration which had increased average bending length (3.23 cm), flexural rigidity (14.88 mg\*cm) and decreased crease recovery angle (114.99°). In 1 and 4 g/l concentrations of softener, few to ~~an~~ average number of microcapsules were present on the treated fabric with poor wash durability. Thus, 2 g/l concentration of the softener was selected as the optimum concentration for further experimental work as it showed ~~presence of~~ many microcapsules with good aroma retention and improved properties of treated wool fabric. ~~Bhatt, 2012~~ suggested that some amount of softener must be added to the padding bath when aroma treatment to fabric was given

using microencapsulated lemongrass essential oil to control the stiffness. The results of the study are also in agreement with study results also agree with Rana *et al.*, 2017 and Pasarkar *et al.*, 2023.

### Optimization of binder concentration

It is evident from the Table 3 and microscopic visualization (Image 3) of treated wool fabric that too many microcapsules were observed on the fabric surface at 10 g/l binder concentration. Wash and wash durability lasted till 25 wash cycles with 3.22 cm average bending length and 14.63 mg\*cm flexural rigidity which was less and 115.66° crease recovery angle which was more as compared to less, and 115.66° crease recovery angle, more than other concentrations of binder binder concentrations, i.e., 5, 15, and 20 g/l. With the increase in binder concentration, it was observed that more number of microcapsules was present on the surface of fabric microcapsules were present on the fabric's surface but that, which was also responsible for the stiffness. It is apparent from the table that a higher number of microcapsules, good wash durability, improved softness, and good resistance to creasing was were found at 10-ten g/l concentration of binder, hence. Hence this was taken as an optimum binder (Beta-cyclodextrin) concentration for application of aroma treatment on wool fabric. These findings are in consistent with Adamowicz, 2015 that the role of binder is to fix the capsules onto the fabric and to consistent with Adamowicz 2015 that the role of a binder is to fix the capsules onto the fabric and hold them in place during wear and washing as it can be chemically bonded or permanently fixed to fabrics. Kumari, 2015 also used 15 g/l binder concentration in the padding bath because at this concentration more number of more microcapsules were deposited on the fabric with good wash durability lasting upto up to 20 wash cycles.

### Optimization of material to liquor ratio

The data in Table 4 and microscopic analysis of aroma-treated aroma-treated wool fabric (Image 4) reveal that at 1:20 M:L ratio, presence of too many microcapsules on the fabric surface was were observed, and wash durability lasted till 25 wash cycles. It was found 1:20 M:L ratio showed improvement in The 1:20 M:L ratio showed improved fabric properties as indicated by decreased average bending length (3.33 cm) and flexural rigidity (15.91 mg\*cm). Also increase in crease recovery angle (114.00°) was noted as compared to other material-to-liquor material-to-liquor ratios of the treatment bath, i.e. 1:30, 1:40, and 1:50. Therefore, on the basis of based on the presence of too many microcapsules on the fabric, good aroma retention, and improved fabric properties, i.e., bending length, flexural rigidity, and crease recovery angle, 1:20 M:L ratio was selected and used for carrying out further research work. The results of the present study are in agreement with Bhatt, 2012 and Kumari, 2015 that the maximum number of microcapsules were present on the fabric at MLR 1:20 with good wash durability and with an increase in MLR, deposition of the number of microcapsules and wash durability of aroma treatment decreased. Similar findings were reported by Krishnaveni, 2021 Krishnaveni, 2021 reported similar findings and El-Molla *et al.*, 2022.

### Optimization of treatment temperature

It is apparent from ~~the~~ Table 5 and microscopic assessment (Image 5) of treated fabrics that at 35 and 45 °C treatment temperatures, too many ~~to many~~ microcapsules were present on the fabric, and wash durability reduced from 25 to 20 wash cycles with increased average bending length from 3.15 to 3.41 cm, flexural rigidity from 14.72 to 16.99 mg\*cm and decreased crease recovery angle from 116.00° to 115.66°. It was further observed that when the treatment temperatures ~~was increased to 55 °C, presence of only~~ increased to 55 °C, only a few microcapsules on the fabric surface with low wash durability was observed. Hence, for carrying out further padding process, 35 °C treatment temperature was optimized as it displayed ~~presence of more number of more~~ microcapsules with good aroma retention and improved fabric properties in terms of softness and resistance to creasing.

### Optimization of treatment time

The data shown in ~~the~~ Table 6 and microscopic evaluation of ~~aroma treated~~ aroma-treated wool fabric (Image 6) indicate that at 20 and 30 minutes treatment duration, too many microcapsules were present on the fabric, and wash durability lasted till 30 wash cycles. But at 30 minutes of treatment time, ~~average bending length (3.03 cm) and flexural rigidity (13.03 mg\*cm) of the treated fabric was less~~ the treated fabric had less average bending length (3.03 cm) and flexural rigidity (13.03 mg\*cm) and. The crease recovery angle (115.33°) was ~~more as compared with the fabric treated for 20 minutes having higher than the fabric treated for 20 minutes, with a~~ 3.24 cm average bending length and 15.56 mg\*cm flexural rigidity with a 115.00° crease recovery angle. Treatment times of 40 and 50 minutes showed ~~presence of only average to few number only average~~ microcapsules on fabric surface with low wash durability, increased average bending length, flexural rigidity and decreased crease recovery angle. Thus, ~~on the basis of based on presence of microcapsules, wash durability and improvement in fabric properties, 30 minutes duration microcapsules, wash durability, and improved fabric properties, 30 minutes~~ was chosen as the optimum time for the aroma treatment of wool fabric with microencapsulated thyme essential oil.

Geethadevi and Maheshwari, 2018 treated bamboo and ~~teneel~~ Tencel fabrics with palmarosa, petitgrain, tea tree, thyme and lavender essential oils for 15 minutes to apply essential oils through pad-dry-cure method. Yuvasri et al., 2020 ~~applied used~~ microcapsules of lemon grass, thyme and lavender essential oils on pure mercerized cotton fabric through the pad-dry-cure method by immersing the fabric samples in microcapsule gel solution for 30 minutes. Naikwadi et al., 2023 used vetiver root extract ~~for the finishing of to finish~~ the organic cotton fabric by pad-dry-cure method, and the treatment temperature was kept at 44 °C.

### Optimization of drying temperature:

It is clear from the Table 7 and through visualization (Image 7) of thyme oil treated fabric samples under a stereo zoom microscope that too many microcapsules were present at 70 °C drying temperature with wash durability lasting till 30 wash cycles having 3.18 cm average bending length, 14.61 mg\*cm flexural rigidity, and 113.50° crease recovery angle. At 60 and 80 °C temperatures, many microcapsules were seen on the fabric and wash durability lasted ~~upto up to~~ 25 wash cycles. At

80 °C temperature, 3.43 cm average bending length, 17.22 mg\*cm flexural rigidity, and 112.00° crease recovery angle was noticed, whereas 3.38 cm average bending length, 15.60 mg\*cm flexural rigidity with 112.66° crease recovery angle was observed at 60 °C drying temperature. However, at 90 °C drying temperature, only a few microcapsules were observed with decreased wash durability (upto 15 wash cycles) and crease recovery angle (111.33°) and increased average bending length (3.46 cm) and flexural rigidity (17.41 mg\*cm). It is thus concluded that at 70 °C drying temperature, soft fabric was obtained with more ~~number of microcapsules as compared to microcapsules thanat~~ other time durations. Therefore, 70 °C was ~~taken as the~~ optimum temperature for drying ~~of aroma treated~~ aroma-treated fabric.

### Optimization of drying time

It can be inferred from Table 8 and microscopic analysis (Image 8) of ~~aroma treated~~ aroma-treated fabric that drying times 2 and 3 minutes exhibited the presence of many microcapsules on the surface of the fabric with wash durability that lasted till 25 wash cycles having 3.32 and 3.43 cm average bending length and 16.21 and 16.89 mg\*cm flexural rigidity with 110.86 and 110.83° crease recovery angle, respectively. At 4 minutes of drying time, too many microcapsules were seen on the fabric surface with a 3.15 cm average bending length, 14.26 mg\*cm flexural rigidity and 113.33° crease recovery angle with wash durability lasting ~~upto up to~~ 30 washes. Whereas, at 5 minutes drying time, an average number of microcapsules were found present on the fabric with wash durability lasting till 15 wash cycles having increased average bending length (3.41 cm) and flexural rigidity (16.81 mg\*cm) and decreased crease recovery angle (109.83°). It is deduced from data in the table that higher number of microcapsules, good wash durability, improved softness, and good resistance to creasing was found at 4 minutes drying time. So, this time duration was selected as optimum drying time for aroma treatment of wool fabric.

Sathianarayanan et al., 2019 applied microencapsulated *tulsi* leaf and pomegranate extract onto the cotton fabric and dried it at 80 °C for 5 minutes. ~~The results of the study are supported by the findings of Thite and Gudiyawar, 2020; Krishnaveni, 2021 and Rana~~ Thite and Gudiyawar, 2020 support the study results; Krishnaveni, 2021; Rana, 2021.

### Optimization of curing temperature

The perusal of the Table 9 indicates that microencapsulated thyme essential ~~oil treated~~ oil-treated fabrics (Image 9) had average bending ~~length-lengths~~ of 3.12, 3.32, 3.35 and 3.39 cm, flexural rigidity of 13.78, 15.85, 16.03 and 17.99 mg\*cm and crease recovery angle of 115.16, 111.83, 111.33, 110.66 degree when cured at 100, 110, 120 and 130 °C, respectively. It is further deduced from the table and microscopic visualization of the microcapsules that too many microcapsules were present on the fabric at temperature of 100 °C having wash durability lasting upto 30 washes with decreased average bending length, flexural rigidity and increased crease recovery angle. Thus, upon a comparison of results of different curing temperatures on ~~different-various~~ parameters, i.e.,

microscopic evaluation of the treated fabrics for the presence of microcapsules, wash durability of aroma, bending length, flexural rigidity and crease recovery angle, **100 °C** temperature was optimized for curing of aroma treated wool fabric.

#### **Optimization of curing time:**

The data in ~~the~~ Table 10 and microscopic images (Image 10) of ~~aroma-treated~~aroma-treated wool fabrics indicate that at curing times 30 and 60 seconds, too many microcapsules were present, having wash durability ~~of aroma~~of smell lasting upto 30 wash cycles, whereas when curing time increased from 90 to 120 seconds, presence of microcapsules was few to very few with wash durability that lasted from 20 to 15 wash cycles. At 60 seconds of curing time, the average bending length (3.06 cm) and flexural rigidity (13.70 **mg\*cm**) of ~~aroma-treated~~aroma-treated fabric were less, while the crease recovery angle (**116.66°**) was observed as more as compared to the other ~~aroma treated~~aroma-treated~~fabrics-materials~~ which were cured for 30, 90 and 120 seconds. Hence, 60 seconds was chosen as the optimum time for ~~curing~~fixing of thyme essential ~~oil-treated~~oil-treated wool fabric as better results in ~~terms of~~ enhanced fabric properties with ~~more~~higher number of durable aroma capsules were obtained at this curing duration.

**Bhatt et al., 2016** applied lemongrass microcapsules onto the cotton fabric by padding ~~process~~and curing of treated fabric was done and curing the treated fabric at **80 °C** for 60 seconds. It was observed that with ~~increase in~~increased curing temperature and time, the walls of microcapsules got ruptured. The results ~~are also in agreement~~also agree with **Sayed et al., 2022** and **Peng et al., 2023**.

### **CONCLUSIONS**

The optimized variables for aroma treatment were found to be 60 g/l microcapsule gel, ~~2~~twog/l ~~softener~~softeners and 10 g/l binder concentration, 1:20 material to liquor ratio, **35 °C** temperature, and 30 minutes treatment time as at these conditions more number of microcapsules, longer wash durability and better fabric properties in terms of bending length, flexural rigidity, and crease recovery angle were observed. ~~Aroma-treated~~Aroma-treated wool fabric, when dried at **70 °C** temperature for 4 minutes and cured at **100 °C** temperature for 60 seconds, exhibited too many microcapsules on the fabric surface with good wash durability, improvement in softness, and good resistance to creasing, hence optimized for drying and curing of microencapsulated thyme essential oil treated wool fabric. The ~~wool fabric being highly hygroscopic in nature~~highly hygroscopic wool fabric is considered a very suitable fabric for the development of aroma textiles. Microencapsulation is ~~found very~~a promising technique that ~~provide~~provides a long-lasting aroma finish by controlling the release rate of microencapsulated thyme essential oil on wool fabric.

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**Table 1: Optimization of microcapsule gel concentration**

Concentration of microcapsules gel (g/l)	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
30	Few	√	√	×	×	×	×	3.32	16.11	112.33	IV
40	Average	√	√	√	×	×	×	3.24	15.33	112.49	III
50	Many	√	√	√	√	×	×	3.25	15.29	114.00	II
<b>60</b>	<b>Many</b>	√	√	√	√	×	×	<b>3.22</b>	<b>14.63</b>	<b>114.99</b>	<b>I</b>

**Table 2: Optimization of softener concentration**

Concentration of softener (g/l)	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
01	Few	√	√	×	×	×	×	3.26	15.46	113.66	IV
<b>02</b>	<b>Many</b>	√	√	√	√	×	×	<b>3.21</b>	<b>14.79</b>	<b>116.83</b>	<b>I</b>
03	Many	√	√	√	√	×	×	3.23	14.88	114.99	II
04	Average	√	√	√	×	×	×	3.26	15.16	112.99	III

**Table 3: Optimization of binder concentration**

Concentration of binder (g/l)	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
05	Average	√	√	√	×	×	×	3.34	16.38	112.66	III
<b>10</b>	<b>Too many</b>	√	√	√	√	√	×	<b>3.22</b>	<b>14.63</b>	<b>115.66</b>	<b>I</b>
15	Many	√	√	√	√	×	×	3.27	15.51	115.33	II

20	Many	√	√	√	√	×	×	3.35	16.40	110.00	IV
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**Table 4: Optimization of MLR of aroma treatment**

MLR	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
1:20	Too many	√	√	√	√	√	×	3.33	15.91	114.00	I
1:30	Many	√	√	√	√	×	×	3.45	16.14	112.99	II
1:40	Few	√	√	√	×	×	×	3.52	16.17	110.33	III
1:50	Few	√	√	√	×	×	×	3.64	16.48	109.66	IV

**Table 5: Optimization of the temperature of aroma treatment**

Treatment temperature (°C)	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
25	Average	√	√	√	×	×	×	3.37	16.68	114.66	III
35	Too many	√	√	√	√	√	×	3.15	14.72	116.00	I
45	Many	√	√	√	√	×	×	3.41	16.99	115.66	II
55	Few	√	√	×	×	×	×	3.47	17.74	114.16	IV

**Table 6: Optimization of time of aroma treatment**

Treatment time (minutes)	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycle)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
20	Too Many	√	√	√	√	√	√	3.24	15.56	115.00	II
30	Too Many	√	√	√	√	√	√	3.03	13.03	115.33	I
40	Average	√	√	√	√	×	×	3.39	16.69	114.83	III
50	Few	√	√	√	×	×	×	3.37	16.85	112.49	IV

**Table 7: Optimization of drying temperature for aroma treatment**

Drying temperature (°C)	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
60	Many	√	√	√	√	√	×	3.38	15.60	112.66	II
70	Too Many	√	√	√	√	√	√	3.18	14.61	113.50	I

80	Many	√	√	√	√	√	×	3.43	17.22	112.00	III
90	Few	√	√	√	×	×	×	3.46	17.41	111.33	IV

**Table 8: Optimization of drying time for aroma treatment**

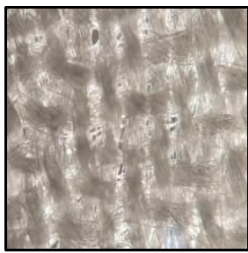
Drying time (minutes)	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
02	Many	√	√	√	√	√	×	3.32	16.21	110.86	II
03	Many	√	√	√	√	√	×	3.43	16.89	110.83	III
<b>04</b>	<b>Too many</b>	√	√	√	√	√	√	<b>3.15</b>	<b>14.26</b>	<b>113.33</b>	<b>I</b>
05	Average	√	√	√	×	×	×	3.41	16.81	109.83	IV

**Table 9: Optimization of curing temperature for aroma treatment**

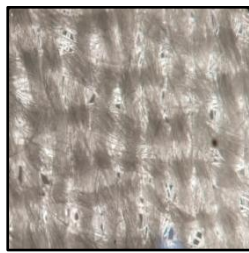
Curing temperature (°C)	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
<b>100</b>	<b>Too Many</b>	√	√	√	√	√	√	<b>3.12</b>	<b>13.78</b>	<b>115.16</b>	<b>I</b>
110	Many	√	√	√	√	√	×	3.32	15.85	111.83	II
120	Few	√	√	√	×	×	×	3.35	16.03	111.33	III
130	Very few	√	√	×	×	×	×	3.39	17.99	110.66	IV

**Table 10: Optimization of curing time for aroma treatment**

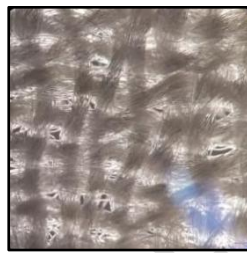
Curing time (seconds)	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycle)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
30	Too many	√	√	√	√	√	√	3.25	15.27	112.83	II
<b>60</b>	<b>Too many</b>	√	√	√	√	√	√	<b>3.06</b>	<b>13.70</b>	<b>116.66</b>	<b>I</b>
90	Few	√	√	√	√	×	×	3.23	15.29	112.33	III
120	Very few	√	√	√	×	×	×	3.28	15.67	111.16	IV



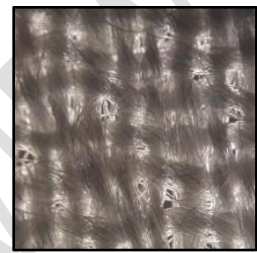
30 g/l (Rank- IV)



40 g/l (Rank- III)



50 g/l (Rank- II)

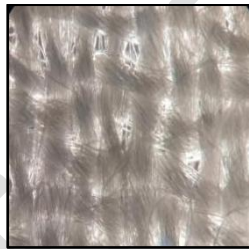


60 g/l (Rank- I)

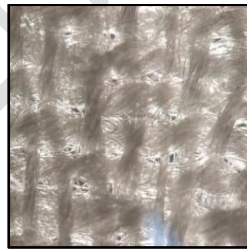
**Image 1: Stereo zoom microscopic images of treated fabric at different concentrations of microcapsule gel**



1.0 g/l (Rank- IV)



2.0 g/l (Rank- I)



3.0 g/l (Rank- II)

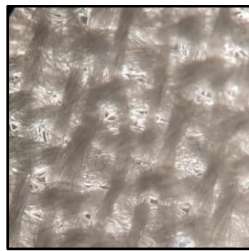


4.0 g/l (Rank- III)

**Image 2: Stereo zoom microscopic images of treated fabric at different concentrations of softener**



5 g/l (Rank- III)



10 g/l (Rank- I)

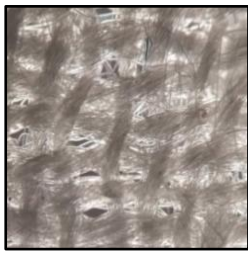


15 g/l (Rank- II)

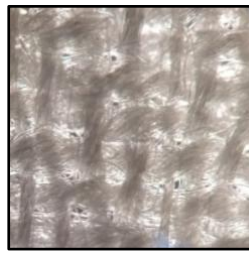


20 g/l (Rank- IV)

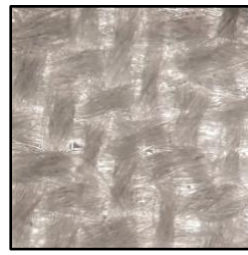
**Image 3: Stereo zoom microscopic images of treated fabric at different concentrations of binder**



1:20 (Rank- I)



1:30 (Rank- II)

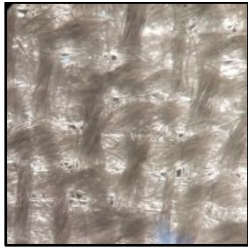


1:40 (Rank- III)

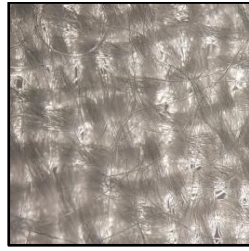


1:50 (Rank- IV)

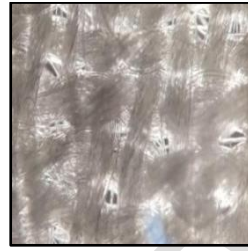
**Image 4: Stereo zoom microscopic images of treated fabric at different MLR**



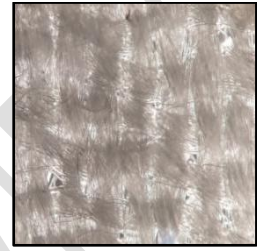
25°C (Rank- III)



35°C (Rank- I)

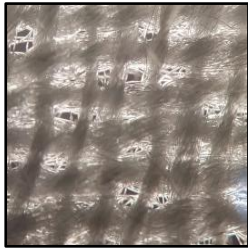


45°C (Rank- II)

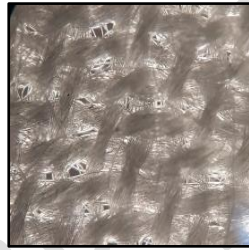


55°C (Rank- IV)

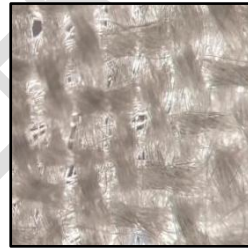
**Image 5: Stereo zoom microscopic images of treated fabric at different treatment temperatures**



20min (Rank- II)



30min (Rank- I)

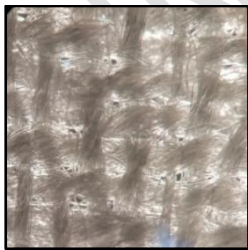


40min (Rank- III)

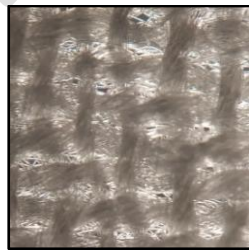


50min (Rank- IV)

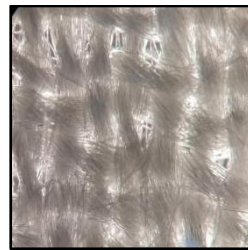
**Image 6: Stereo zoom microscopic images of treated fabric at different treatment times**



60°C (Rank- II)



70°C (Rank- I)

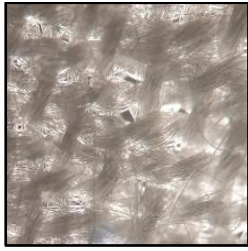


80°C (Rank- III)

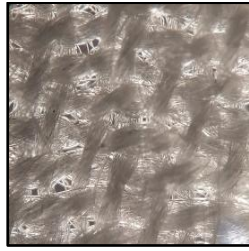


90°C (Rank- IV)

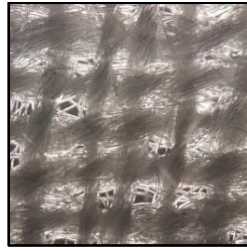
**Image 7: Stereo zoom microscopic images of treated fabric at different drying temperatures**



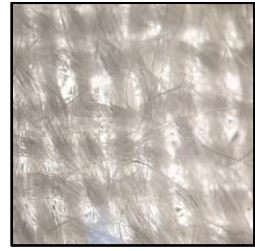
2 min (Rank- II)



3 min (Rank- III)

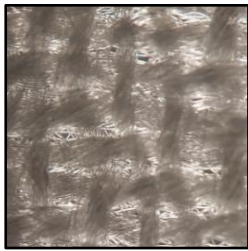


4 min (Rank- I)

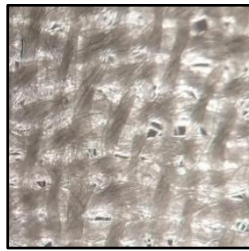


5 min (Rank- IV)

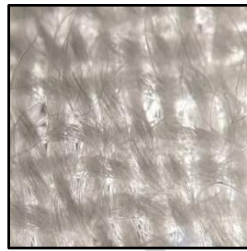
**Image 8: Stereo zoom microscopic images of treated fabric at different drying times**



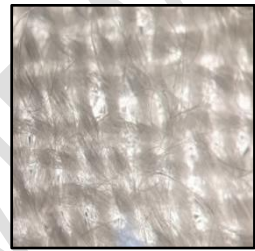
100°C (Rank- I)



110°C (Rank- II)

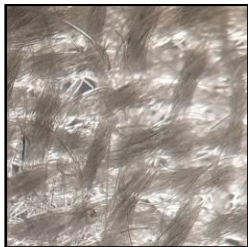


120°C (Rank- III)

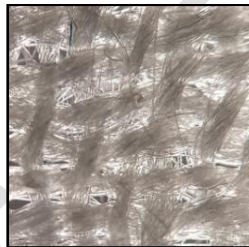


130°C (Rank- IV)

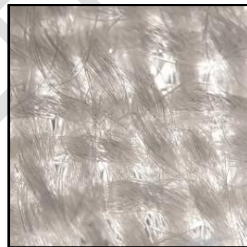
**Image 9: Stereo zoom microscopic images of treated fabric at different curing temperatures**



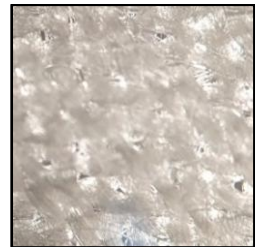
30 sec (Rank- II)



60 sec (Rank- I)



90 sec (Rank- III)



120 sec (Rank- IV)

**Image 10: Stereo zoom microscopic images of treated fabric at different curing times**