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

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Background: With rising global trends and changing lifestyles in fashion, beauty as well as healthcare, the awareness of consumers has enforced the evolution of specialty value-added textiles. Essential oils and basic aromatherapy ingredients are microencapsulated and applied to the materials to provide a therapeutic effect and 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 based on its aromatic and therapeutic properties. A complex coacervation technique of microencapsulation was used to prepare thyme oil microcapsules. The padding bath components and treatment variables were optimized based on

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the presence of microcapsules on wool fabric as observed under a stereo zoom microscope, aroma durability to washing and improved properties of treated material regarding bending length, flexural rigidity and crease recovery angle. The aroma treatment was given on wool fabric using pad-dry-cure method.

Results: Optimized variables for aroma treatment were 60 g/l microcapsule gel, two g/l softeners and ten g/l binder concentration, 1:20 material to liquor ratio, 35 ºC 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 wool fabric were dried at 70 ºC temperature for 4 minutes and cured at 100 ºC for 60 seconds.

Conclusions: Thyme essential oil has many reported therapeutic properties therefore microencapsulated thyme basic oil-treated wool fabric can be used for apparel, home, and healthcare textiles. Higher 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 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.

Keywords: Thyme essential oil microcapsules; wool fabric; optimization; pad-dry-cure method; aroma treatment.

1. INTRODUCTION

A consumer-oriented 21st century challenges garment and fabric manufacturers 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 fresh ingredients on the fabric to provide functional benefits to the end product West and Annett-Hitchcock, [1]; Eyupoglu et al. [2]. Available finishes from the natural substrates 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 for finishing application due to their efficacy without harmful effects Naikwadi et al. [3]; Sayed et al. [4].

Fragrances in essential oils and aromatic compounds, when applied to textile materials, give the textile a pleasant odor that 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 and the beneficial effect of aromatherapy. Aromatherapy does not cure conditions but helps the body to find a natural way to heal itself and improves immune response Kumar et al. [5]; Sousa et al. [6]. Aromatic oily liquids, 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. Various essential oils are used in aromatherapy for moisturizing, refreshing, and other wellness effects Ahmad et al. [7]; Hamid et al. [8].

The thyme essential oil is a combination of monoterpenes. The primary substance of this oil is phenol isomer carvacrol, and it has active biological actions such as antifungal, antibacterial, antioxidant activity, antinatalism, and antispasmodic. Thyme is commonly used as a culinary herb and for different medicinal purposes. Nowadays, thyme presents various functional possibilities in the pharmacy, food, and cosmetic industries. The interest in formulating pharmaceuticals, nutraceuticals, and cosmeceuticals based on thyme is due to treating disorders affecting the respiratory, nervous, and cardiovascular systems. Thyme essential oil-treated fabric creates a microclimate for the wearer. It controls the release of essential oil through friction and body movement, which will play a significant role in the health and wellness of the wearer Salehi et al. [9]; Silva et al. [10].

Textile materials are treated with pleasant odor-producing essential oils and aromatic compounds to impart an aroma finish so the wearer gets some valuable effects. Due to their highly volatile nature, these are ineffective for profitable textile applications. But microencapsulation technology locks essential oils with 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 micrometer to millimeter range for protection and/or later release. The unique advantage of microencapsulation 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 preparation methods are chosen Zhao et al*.* [11]; Rukhaya et al*.* [12].

As close friends of humans, textiles can make aromatherapy easy wherever these are needed. Among all the natural origin fibers, wool plays a significant role in the textile industry. Wool is a crude protein fiber, and the wool polymer is a linear keratin polymer. Its repeating unit is sulfur-containing amino acids linked with disulfide bonds Srivastava and Srivastava, [13]; Rukhaya et al*.* [14]. Wool is a very absorbent fiber as it has higher amorphous areas, i.e., about 75-70 percent amorphous and 25-30 percent crystalline; however, the scaly structure of wool makes it partially water repellent, but when moisture or other substances like oil and aroma once penetrate the fiber surface, get absorbed quickly and has good retention for a longer time.

2. MATERIALS AND METHODS

2.1 Materials

Pure greige woven wool fabric having 59.40 ends/in (EPI), 49.00 picks/inch (PPI), 161 g/m^2 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 (βeta-cyclodextrin), were provided by chemical suppliers in Haryana, India. Other materials, such as acetic acid, formalin, sodium hydroxide, and a wetting agent (Ultravon JU), were also used in the study.

2.2 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 ratio 1:20 and pH 7. The fabric was added to the scouring bath, and the temperature was raised gradually and held at 60 ºC. 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.

2.3 Preparation of Thyme Oil Microcapsules

The phase separation-complex coacervation technique of microencapsulation was used to prepare thyme essential oil microcapsules. 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 based on the size, distribution, and quality of the wall of capsules; different process variables were optimized; 16 g of gelatin was weighed and dissolved in 25 ml warm water and stirred using a high-speed stirrer for 10 minutes; 4 g of vetiver oil was added to the solution at 45 ºC; 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 ºC. The pH of the solution was decreased to 4.5 by adding dilute acetic acid and 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.

2.4 Standardization of Aroma Treatment for Wool Fabric

For aroma treatment, 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 ratio, treatment temperature and time, drying temperature and time, curing temperature and time, were optimized 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 crease recovery angle. The aroma treatment was given to wool fabric using optimized concentrations and conditions of the pad-dry-cure finish application method.

2.5 Optimization of Padding Bath Components

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

i. Optimization of microcapsule gel concentration: To determine the optimum concentration of microcapsule gel, four padding 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 ºC for 30 minutes with occasional stirring. Afterward, the fabric was placed on the trough of the padding mangle with padding solution and passed through the squeezing rollers of the padding mangle at pneumatic pressure of 2 kg/cm^2 with two dips and nips having 80-90 percent expression. As the fabric left the padding mangle, it was dried at 80 ºC for 5 minutes and cured at 110 ºC for 1 minute Thilagavathi et al*.* [15]; Kumari, [16].

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

iii. Optimization of binder concentration: For optimization of 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 were carried out, and binder concentration optimization was done.

2.6 Optimization of Material-to-Liquor Ratio

To determine the optimum material-to-liquor ratio (MLR) of the padding bath, four different 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. At the same time, other variables of the pad-dry-cure method were kept constant. The optimum M:L ratio was selected for the presence of microcapsules, wash durability, and improved properties of treated fabric, i.e., bending length, flexural rigidity, and crease recovery angle.

2.7 Optimization of Treatment Temperature

The aroma treatment was given to wool fabric at four different temperatures, i.e., 25, 35, 45, and 55ºC with optimized concentrations (microcapsule gel, softener, and binder) and conditions (M:L ratio) while other variables were kept constant. The fabric's padding, drying and curing were carried out, and the temperature exhibiting the best results was selected as the optimum treatment temperature.

2.8 Optimization of Treatment Time

The treatments were carried out for four different time durations, 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. Based on 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.

2.9 Optimization of Drying Temperature

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

2.10 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.

2.11 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 130ºC, keeping fixing time constant and optimization of curing temperature was done.

2.12 Optimization of Curing Time

After applying aroma treatment, the padded samples were dried at optimum temperature and time and cured at optimum temperature for four different curing times, i.e., 30, 60, 90, and 120 seconds. 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.

3. RESULTS AND DISCUSSION

3.1 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 based on the presence of microcapsules on the treated wool fabric, good aroma retention to washing, and improved properties of the treated fabric.

3.2 Optimization of Microcapsule Gel Concentration

The data in Table 1 and microscopic assessment of the 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. The degree of crease recovery angle was less (114.00º) 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 (114.99º). At other concentrations, the presence of few to average microcapsules was seen on the fabric surface with low aroma retention. It is evident from the table that more 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 the padding bath. Thite and Gudiyawar, [17] also used the ratio 50:50 of microcapsule gel and water to prepare the padding bath for applying *tulsi*, lemongrass, and citronella essential oil on woven cotton fabric. Similar results were reported by Rana et al*.* [18]; Lim and Setthayanond, [19].

3.3 Optimization of Softener Concentration

It is evident from the Table 2 and microscopic evaluation (Image 2) of the thyme essential 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 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 116.83º 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 (114.99º). 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 many microcapsules with good aroma retention and improved properties of treated wool fabric. Bhatt, [20] 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 study results also agree with Rana et al*.* [18] and Pasarkar et al*.* [21].

3.4 Optimization of Binder Concentration

It is evident from 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 durability lasted 25 wash cycles with 3.22 cm average bending length and 14.63 mg*cm flexural rigidity less, and 115.66º crease recovery angle, more than other binder concentrations, i.e., 5, 15, and 20 g/l. With the increase in binder concentration, it was observed that more microcapsules were present on the fabric's surface, 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 were found at ten g/l concentration of binder. Hence this was taken as an optimum binder (Beta-cyclodextrin) concentration for application of aroma treatment on wool fabric. These findings are consistent with Adamowicz [22] 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, [16] also used 15 g/l binder concentration in the padding bath because more microcapsules were deposited on the fabric with good wash durability lasting up to 20 wash cycles.

3.5 Optimization of Material to Liquor Ratio

The data in Table 4 and microscopic analysis of aroma-treated wool fabric (Image 4) reveal that at 1:20 M:L ratio, too many microcapsules on the fabric surface were observed, and wash durability lasted till 25 wash cycles. 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 (114.00º) was noted as compared to other material-to-liquor ratios of the treatment bath, i.e. 1:30, 1:40, and 1:50. Therefore, 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, [20] and Kumari, [16] 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. Krishnaveni, [23] reported similar findings and El-Molla et al. [24].

3.6 Optimization of Treatment Temperature

It is apparent from Table 5 and microscopic assessment (Image 5) of treated fabrics that at 35 and 45 ºC treatment temperatures, too 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 116.00º to 115.66º. It was further observed that when the treatment temperatures increased to 55 ºC, only a few microcapsules on the fabric surface with low wash durability was observed. Hence, for carrying out further padding process, 35 ºC treatment temperature was optimized as it displayed more microcapsules with good aroma retention and improved fabric properties in terms of softness and resistance to creasing.

3.7 Optimization of Treatment Time

The data shown in Table 6 and microscopic evaluation of 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, the treated fabric had less average bending length (3.03 cm) and flexural rigidity (13.03 mg*cm). The crease recovery angle (115.33º) was 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 115.00º crease recovery angle. Treatment times of 40 and 50 minutes showed only average microcapsules on fabric surface with low wash durability, increased average bending length, flexural rigidity and decreased crease recovery angle. Thus, based on 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, [25] treated bamboo and Tencel fabrics with palmarosa, petitgrain, tea tree, thyme and lavender essential oils for 15 minutes to apply essential oils through pad-dry-cry method. Yuvasri et al. [26] 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. [27] used vetiver root extract to finish the organic cotton fabric by pad-dry-cure method, and the treatment temperature was kept at 44 º C.

3.8 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 ºC drying temperature with wash durability lasting till 30 wash cycles having 3.18 cm average bending length, 14.61 mg*cm flexural rigidity, and 113.50º crease recovery angle. At 60 and 80 ºC temperatures, many microcapsules were seen on the fabric and wash durability lasted up to 25 wash cycles. At 80 ºC temperature, 3.43 cm average bending length, 17.22 mg*cm flexural rigidity, and 112.00º crease recovery angle was noticed, whereas 3.38 cm average bending length, 15.60 mg*cm flexural rigidity with 112.66º crease recovery angle was observed at 60ºC drying temperature. However, at 90ºC drying temperature, only a few microcapsules were observed with decreased wash durability (upto 15 wash cycles) and crease recovery angle (111.33º) and increased average bending length (3.46 cm) and flexural rigidity (17.41 mg*cm). It is thus concluded that at 70ºC drying temperature, soft fabric was obtained with more microcapsules than at other time durations. Therefore, 70 ºC was the optimum temperature for drying aroma-treated fabric.

3.9 Optimization of Drying Time

It can be inferred from Table 8 and microscopic analysis (Image 8) of 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 110.83º 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 113.33º crease recovery angle with wash durability lasting 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 (109.83º). 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*.* [28] applied microencapsulated *tulsi* leaf and pomegranate extract onto the cotton fabric and dried it at 80ºC for 5 minutes. Thite and Gudiyawar, [17] support the study results; Krishnaveni, [23]; Rana, [29].

3.10 Optimization of Curing Temperature

The perusal of the Table 9 indicates that microencapsulated thyme essential oil-treated fabrics (Image 9) had average bending 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 ºC, 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 ºC 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 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 ºC temperature was optimized for curing of aroma treated wool fabric.

3.11 Optimization of Curing Time

The data in Table 10 and microscopic images (Image 10) of aroma-treated wool fabrics indicate that at curing times 30 and 60 seconds, too many microcapsules were present, having wash durability of smell lasting up to 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 fabric were less, while the crease recovery angle (116.66º) was observed as more as compared to the other aroma-treated materials which were cured for 30, 90 and 120 seconds. Hence, 60 seconds was chosen as the optimum time for fixing thyme essential oil-treated wool fabric as better results in enhanced fabric properties with more durable aroma capsules were obtained at this curing duration.

Bhatt et al. [30] applied lemongrass microcapsules onto the cotton fabric by padding and curing the treated fabric at 80 ºC for 60 seconds. It was observed that with increased curing temperature and time, the walls of microcapsules got ruptured. The results also agree with Sayed et al*.* [4]; Peng et al*.* [31].

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
60	Many	√	√	√	√	×	×	3.22	14.63	114.99	I

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
02	Many	√	√	√	√	×	×	3.21	14.79	116.83	I
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
10	Too many	√	√	√	√	√	×	3.22	14.63	115.66	I
15	Many	√	√	√	√	×	×	3.27	15.51	115.33	II
20	Many	√	√	√	√	×	×	3.35	16.40	110.00	IV

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 (^{o}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

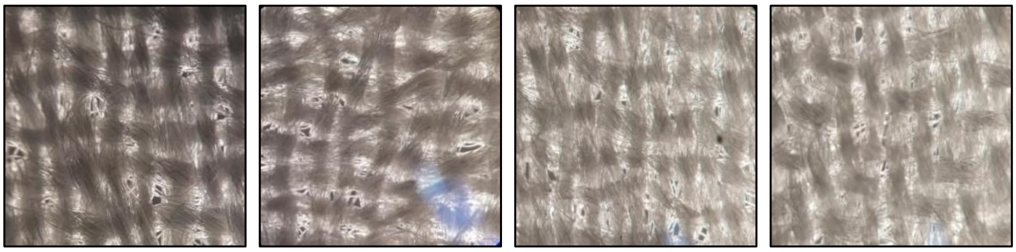
Drying time (minutes)	Parameters										Ranks
	Presence of microcapsules on fabric	Wash durability (Wash cycles)						Average bending length (cm)	Flexural rigidity (mg-cm)	Average crease recovery (Degree)	
		5	10	15	20	25	30				
02	Many	√	√	√	√	√	×	3.32	16.21	110.86	II
03	Many	√	√	√	√	√	×	3.43	16.89	110.83	III
04	Too many	√	√	√	√	√	√	3.15	14.26	113.33	I
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				
100	Too Many	√	√	√	√	√	√	3.12	13.78	115.16	I
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

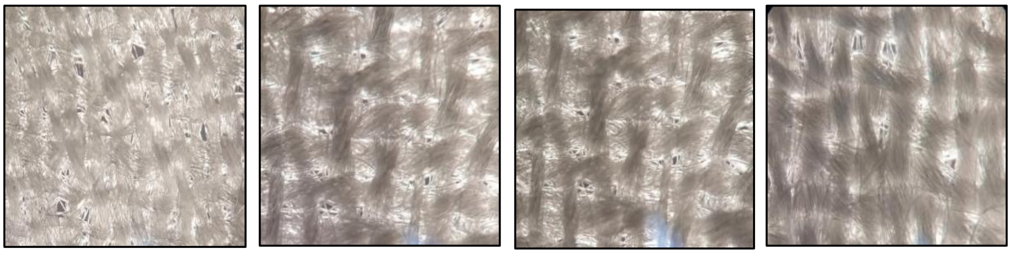
Table 10. Optimization of curing time for aroma treatment

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
60	Too many	√	√	√	√	√	√	3.06	13.70	116.66	I
90	Few	√	√	√	√	×	×	3.23	15.29	112.33	III
120	Very few	√	√	√	×	×	×	3.28	15.67	111.16	IV



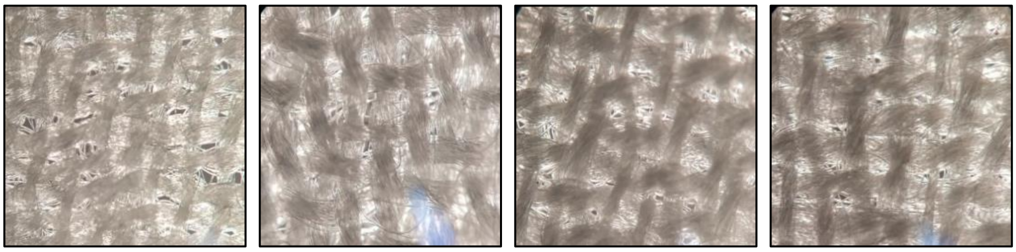
30 g/l (Rank- IV) 40 g/l (Rank- III) 50 g/l (Rank- II) 60 g/l (Rank- I)

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



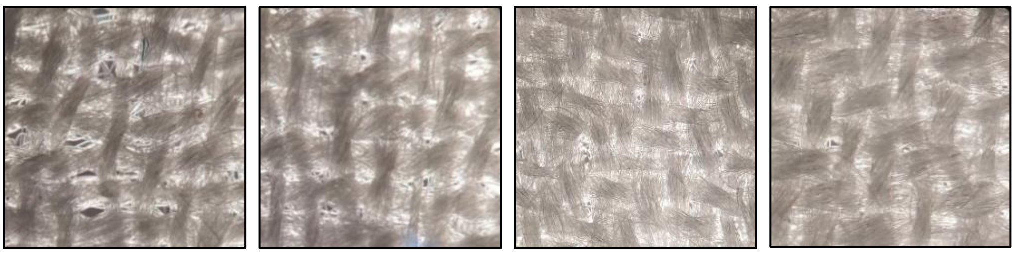
1.0 g/l (Rank- IV) 2.0 g/l (Rank- I) 3.0 g/l (Rank- II) 4.0 g/l (Rank- III)

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



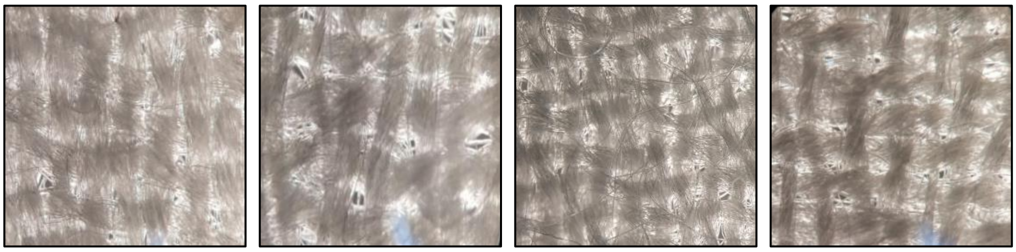
5 g/l (Rank- III) 10 g/l (Rank- I) 15 g/l (Rank- II) 20 g/l (Rank- IV)

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



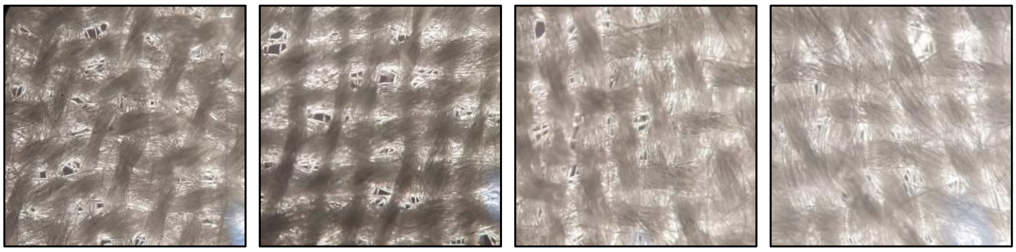
1:20 (Rank- I) 1:30 (Rank- II) 1:40 (Rank- III) 1:50 (Rank- IV)

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



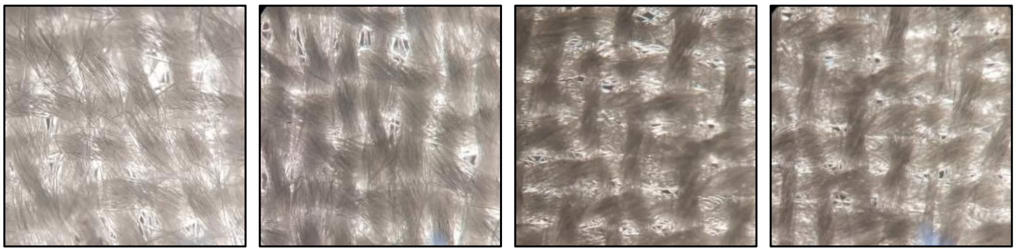
25^{o}C (Rank- III) 35^{o}C (Rank- I) 45^{o}C (Rank- II) 55^{o}C (Rank- IV)

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



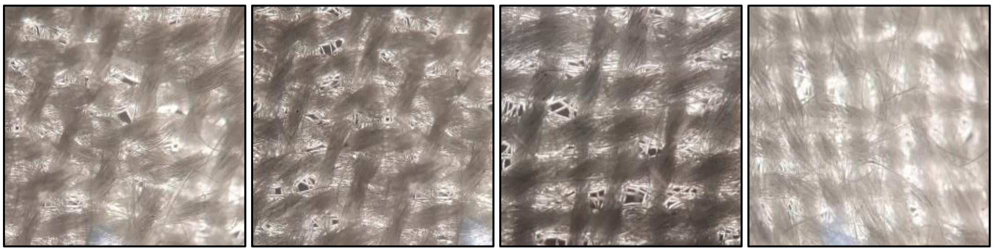
20 min (Rank- II) 30 min (Rank- I) 40 min (Rank- III) 50 min (Rank- IV)

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



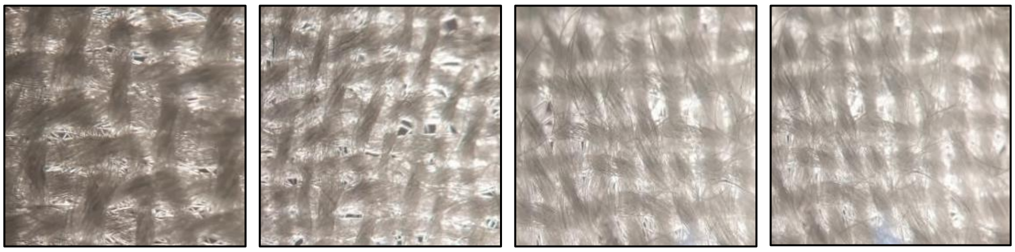
60^{o}C (Rank- II) 70^{o}C (Rank- I) 80^{o}C (Rank- III) 90^{o}C (Rank- IV)

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



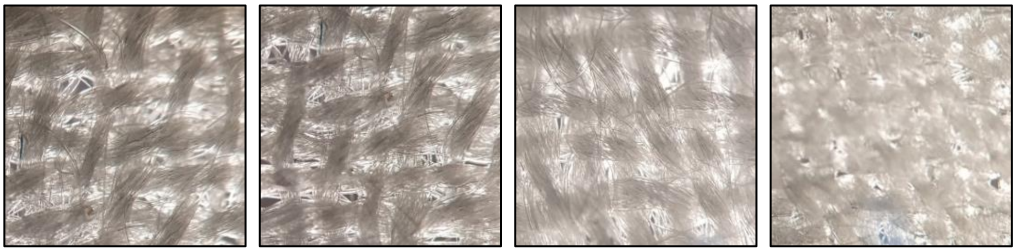
2 min (Rank- II) 3 min (Rank- III) 4 min (Rank- I) 5 min (Rank- IV)

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



100^{o}C (Rank- I) 110^{o}C (Rank- II) 120^{o}C (Rank- III) 130^{o}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

4. CONCLUSIONS

The optimized variables for aroma treatment were found to be 60 g/l microcapsule gel, two g/l softeners and 10 g/l binder concentration, 1:20 material to liquor ratio, 35 ºC 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 wool fabric, when dried at 70 ºC temperature for 4 minutes and cured at 100 ºC 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 highly hygroscopic wool fabric is considered a very suitable fabric for the development of aroma textiles. Microencapsulation is a promising technique that provides a long-lasting aroma finish by controlling the release rate of microencapsulated thyme essential oil on wool fabric.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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