Durable Aroma Finishing of Wool Fabric with Microencapsulated Vetiver Essential Oil and Assessment of its Properties

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ABSTRACT

The present study was conducted to develop aroma wool fabric using microencapsulated vetiver essential oil without deteriorating the fabric properties. SEM analysis of the treated wool fabric depicted adhesion of numerous microcapsules of spherical shape and FTIR analysis indicated presence of different functional groups on the fabric. The aroma treatment improved most of the tested physical properties of wool fabric. Aroma treated fabric displayed good antimoth efficacy in terms of weight reduction (7.57%) and moth mortality (40%). Treated wool fabric also exhibited antibacterial activity with 63.45 and 61.37 percent reduction in bacterial growth against S. aureus and E.coli, respectively. The UPF value of scoured fabric (30.25) enhanced to 87.70 after treatment with microencapsulated vetiver oil. Aroma durability in terms of retention and intensity of aroma in treated fabric was assessed and good aroma durability was noticed against washing, abrasion, ironing and sun-drying. Hence, microencapsulated vetiver oil appears to be an appropriate aspirant for the development of multifunctional textiles having fragrance with added antimoth, antibacterial and UV protection properties.

KEYWORDS: *Vetiver oil, Microencapsulation,Wool fabric, Aroma treatment, Physical and Functional Properties, Aroma durability.*

1. INTRODUCTION

The increase in the market competitiveness along with the diversity of consumers' demands has created a challenging environment in the textile sector. Therefore, the textile industry has shown a growing interest in the finishing of conventional fabrics to produce innovative products with durable functional properties that

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enhance health, safety and ergonomics by employing green chemistry materials and processes[24]. Aroma finish is a process by which the textile materials are infused with bioactive systems (e.g., gum acacia/essential oil, chitosan/essential oil) that finally get the multifunctional properties and a feeling of well-being and freshness in the wearer[10].

Essential oils which are basic ingredients of aromatherapy derived from plants are composed of natural aromatic molecules endowed with many physiological as well as therapeutic properties[9,37]. Vetiver oil is one of the essential oils that can be used as an eco-friendly finishing agent to impart functional properties to textiles. Vetiver grass also known by the Latin name *Vetiveria zizanioides* or local name *'Khus'*, a part of the *Poaceae* family, is a tall, tufted, perennial, scented grass, with a straight stem, long narrow leaves and a lacework root system. The root is the most valuable part of the grass as it contains the majority of the essential oil[5,27]. It has been traditionally used in aromatherapy from a long period of time for relieving stress, anxiety, depression, nervous tension and insomnia [14].

One of the major limitations in the natural material based aroma finishing is the non-durability of the finish, since they cannot form any bond with the textile materials. Microencapsulation of natural materials is one of the methods used to increase the durability of the finish in which the active compounds are encapsulated using a wall material like sodium alginate, gum acacia etc. and applied on the textile materials[33]. Microencapsulation can effectively control the release rate of fragrance compounds which ensures the storage life of volatile substance. Release of aroma is by diffusion of the fragrance through the capsule wall and rupture of the capsules[34].

As close friends of human, textiles can make aromatherapy easy wherever these are needed. Among all the natural origin fibres, wool plays a significant role in textile industry. Wool is a natural protein fibre and wool polymer is a linear keratin polymer. Its repeating unit is sulphur containing amino acid linked together with disulphide bonds. Wool forms hydrogen bond between hydrogen and nitrogen atom which is very weak and breaks easily when fibre absorbs water that penetrates easily in fibre polymer system [28]. Wool is very absorbent fibre as it contains higher amorphous than crystalline areas, 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 surface, get absorbed quickly and has good retention for a longer time [29]. Hence, the present study was planned in order to exploit the aromatic and therapeutic properties of vetiver essential oil and its application on wool fabric with the objectives to evaluate the effect of microencapsulated vetiver essential oil on properties of treated wool fabric and to assess the durability of aroma treatment on the treated fabric.

2. EXPERIMENTAL

2.1 Materials

Pure griege woven wool fabric with 59 ends/inch (EPI), 49 picks/inch (PPI), 162 g/m^2 basis weight having 0.3 mm thickness was procured from market of Ludhiana city of Punjab, India. Vetiver essential oil was provided by Emmbros Overseas Lifestyle Pvt. Ltd., Haryana, India. Wall materials (gum acacia and gelatin), softener (silicon) and binder (βeta-cyclodextrin) were purchased from chemical suppliers of Haryana, India. Acetic acid, formalin, sodium hydroxide and wetting agent (Ultravon JU) were also used in the study.

2.2 Preparation of Fabric

The wool fabric was scoured using neutral wetting agent (2 g/l) keeping material to liquor ratio 1:20 and pH 7 at 60°C temperature for 60 minutes with occasional stirring. The scoured fabric was used as a control and reference. The physical and functional properties of scoured wool fabric were measured and used for comparison with aroma treated wool fabric.

2.3 Preparation of Vetiver Oil Microcapsules

Complex coacervation technique was used for preparing microcapsules. Experiments were conducted to optimize various variables of microcapsule gel formation *i.e.* ratios of essential oil:gum:gelatin, temperature and pH to obtain the desired results. The microencapsulation process was optimized on the basis of visual evaluation through inverted microscope on three parameters i.e. size of microcapsules, uniformity in size and distribution and walls of microcapsules. 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°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 at high speed for 20 minutes. The pH of the solution was increased to 7.0 using sodium hydroxide solution to form microcapsule gel. For stabilization, 1 ml of 17 percent alcoholic formalin was added to the formed capsules[17]. The microcapsules were then applied on the wool fabric.

2.4 Application of Microcapsules on Wool Fabric

Experiments were performed to standardize the process of aroma treatment for application of microencapsulated vetiver oil on wool fabric through pad-dry-cure method. On the basis of presence of microcapsules on the fabric observed under stereo zoom microscope, longer wash durability and improvement in fabric properties i.e. bending length, flexural rigidity and crease recovery angle, different concentrations and conditions were optimized. The scoured wool fabric was first impregnated in padding solution containing optimum concentrations of microcapsule gel, softener and binder i.e. 60 g/l, 2 g/l and 10 g/l, respectively keeping 1:20 material to liquor ratio and aroma treatment was given for optimum 30 minutes at 35°C temperature. The impregnated fabric was passed between the rollers of the padding mangle maintaining the pneumatic pressure of 2 kg/cm^2 with two dips and nips having 80-90 percent expression. Further the fabric was dried in a hot air oven using optimum conditions i.e. 70°C temperature for 4 minutes and finally cured at optimum 100°C temperature for 60 seconds.

2.5 Characterization

Scanning Electron Microscope (SEM) model Philips XL-30 was used to investigate the morphology of treated fabric at an accelerating voltage of 10 KV with the resolution of 20 µm and magnification ranging from 800-1000X. The treated fabrics of 10 mm diameter were mounted on the circular metallic sample holder, kept inside the SEM instrument, observed at different magnification and images were captured. The magnified images were used to confirm the presence of microcapsules on the wool fabric. The characterization in terms of interactions and chemical absorption of scoured and treated fabrics was determined through Fourier Transform Infrared Spectroscopy. The fabrics were chopped to powder and placed at the FTIR instrument. The transmittance spectrum was recorded from 800-3800cm^{-1}.

2.6 Testing of Physical Properties

The preliminary properties of scoured and treated wool fabrics were tested for three parameters *i.e.* fabric count, weight and thickness. Pick glass with pointer was used to determine the fabric count of the wool fabric using ASTM-D123 test method. The weight of one square meter of the fabric was calculated as per ASTM-D3776-90 test method by multiplying the weight of specimen in grams with 100. Digital thickness tester was used to measure thickness of the fabrics using ASTM-D 1777-60 test method.

Mechanical properties *viz.* bending length, flexural rigidity, tensile strength and elongation were assessed.

Stiffness Tester was used to determine the bending length and flexural rigidity of fabrics as per ASTM-D 1388-64 test method. Flexural rigidity was measured using equation-1 and 2.

Flexural rigidity in warp and weft direction

$$(G_1 \text{ and } G_2)\ W \times C^2 \text{mg} - \text{cm} \qquad (1)$$

Where, W is the weight per unit area of the fabric in milligrams per square centimeter; C is the mean bending length for the respective direction.

$$\text{Overall Flexural Rigidity } (G) = \sqrt{G_1 \times G_2} \qquad (2)$$

Digital Tensile Strength Tester was used to determine the tensile strength and elongation of the scoured and treated fabrics, according to IS: 4169 standard test method.

Performance properties *i.e.* moisture regain, wickability, water absorbency, crease recovery angle and air permeability were also measured. BS1051:1960 test method was used to determine the moisture regain. The wickability was determined using AATCC 1945 standard test method. Water absorbency of the scoured and treated fabrics was measured to test the propensity of the fabric to take up water. Drop test was performed to measure the water absorbency of control and treated fabrics. The crease recovery performance of fabrics was measured using BS3086:1972 test method on the Crease Recovery Tester. Air permeability of fabrics was measured using Air Permeability Tester based on Kawa bata KES-F8-API according to ASTM D737 test method. The five readings averaged to determine all these properties and analyzed statistically using the paired t-test.

2.7 Testing of Functional properties

The scoured and aroma treated fabrics were measured for antimoth property. The samples of size 1.5×5 cm were taken from both fabrics, conditioned under standard atmosphere and kept in poly petri dishes separately along with ten alive adult carpet beetle moths. Petri dishes were kept in incubator for 15 days at temperature between 30-35°C with 50-65 percent relative humidity. After 15 days, the number of moths, alive and dead was counted. Then all the moths were taken out and the samples were cleaned and conditioned for 24 hours. The fabrics were weighed again and the percent weight loss was calculated[15]. The fabrics were also evaluated for antibacterial property using standard quantitative test method *i.e.* AATCC Test Method 100. Zone of inhibition, CFU (Colony Forming Unit) and percent reduction in the bacterial count were calculated. UPF of scoured and treated fabrics was determined according to test method UVR TRANSMISSION AATCC-183:2004 using SDL UV penetration and protection measurement system (Compsec M 350 UV- Visible Spectrometer). The UV protection category was determined by the UPF values described by Australia/ New Zealand Standards AS/ NZS4399 (1996).

2.8 Assessment of Aroma Durability

2.8.1 Retention of Aroma

The aroma treated wool fabrics were got assessed qualitatively for aroma retention from 25 experts using olfactory analysis after exposing to different agencies *i.e.* washing, abrasion, ironing and sun-drying. A portion of each treated fabric (3x3 cm) was taken out and tested for the presence of aroma by experts. Prior to being judged, the samples were hung on a clothesline in a room for 1 hour to stabilize the evaporation of fragrance. To get fair judgement the experts were not allowed to enter the stabilizing room and samples were brought to the experts. To detect odour, 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 responses were recorded as 'Yes' or 'No' [16].

2.8.2 Intensity of Aroma

The treated fabrics were also evaluated for intensity of aroma. Change in intensity of aroma was assessed by sensory evaluation from 25 experts. The assessment was done on five point continuum scale with rating as very strong (5), strong (4), moderate (3), mild (2) and faint (1). The aroma was assessed after different washing cycles (5, 10, 15, 20, 25 and 30), abrasion cycles (10, 50 and 100), number of times of ironing (1, 5 and 10) and number of sun-drying hours (1, 2 and 3) to test the effect of these conditions on aroma performance. The experimental data was analyzed by using frequencies, percentage and weighted mean scores (WMS).

3. RESULTS AND DISCUSSION

3.1 Characterization of Aroma Treated Wool Fabric through SEM Analysis

The Scanning Electron Microscopic analysis of the scoured and treated fabrics was done to analyze any morphological changes that occurred due to aroma treatment. The treated fabric surface was coated with numerous microcapsules containing vetiver essential oil inside the matrix which were spherical in shape with relatively uniform size as observed from the Fig. 1. The results are in agreement with earlier reports of Ali *et al.*[1], Bhatt *et al.*[4], Petrulyte *et al.*[21] and Wijesirigunawardana and Perera[38] that too many microcapsules of spherical shape were found adhered on the surface of aroma treated fabric.

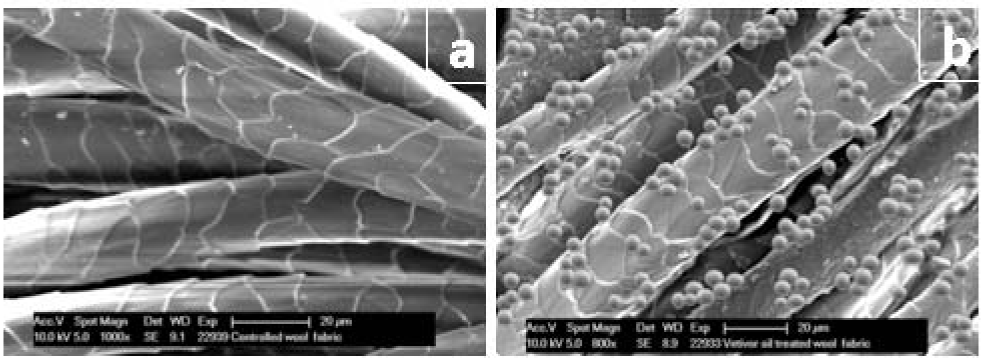

Figure 1. SEM images of wool fabric (a) scoured and (b) treated

3.2 Characterization of Aroma Treated Wool Fabric through FTIR Analysis

Fig. 2 depicts the presence of different functional groups i.e. C-N stretch primary amine, C-O stretch alkyl aryl ether, C-H bend methine, N-O stretch nitro compound, C=C/C=O cyclic alkene/amide, C-H methine and O-H/N-H stretch alcohol/amine in scoured and treated fabrics. After aroma treatment, there was a slight shift in peaks from 1066 to 1022,1235 to 1259, 1451 to 1448, 1517 to 1518, 1628 to 1625, 2936 to 2962 and 3281 to 3278 cm^{-1} in treated wool fabric which were considered responsible for change in properties. Biswas *et al.*[6] also noticed that there was a slight shift in band/peaks from untreated to aroma treated fabric.

3.3 Effect of Aroma Treatment on Preliminary and Mechanical Properties of Wool Fabric

Table 1 presents the preliminary properties of scoured and treated wool fabrics. The fabric count of scoured fabric was 55×45 (ends and picks per inch) which increased to 62×52 in treated fabric. It can also be observed from the table that mean fabric weight and thickness increased after aroma treatment. Statistically, a significant difference ($p<0.05$) was observed in fabric count and weight of treated fabrics. The increment in preliminary properties may be attributed to the fact that application of aroma finish caused shrinkage and brought warp and weft yarns closer to each other. It may also be due to the deposition of numerous

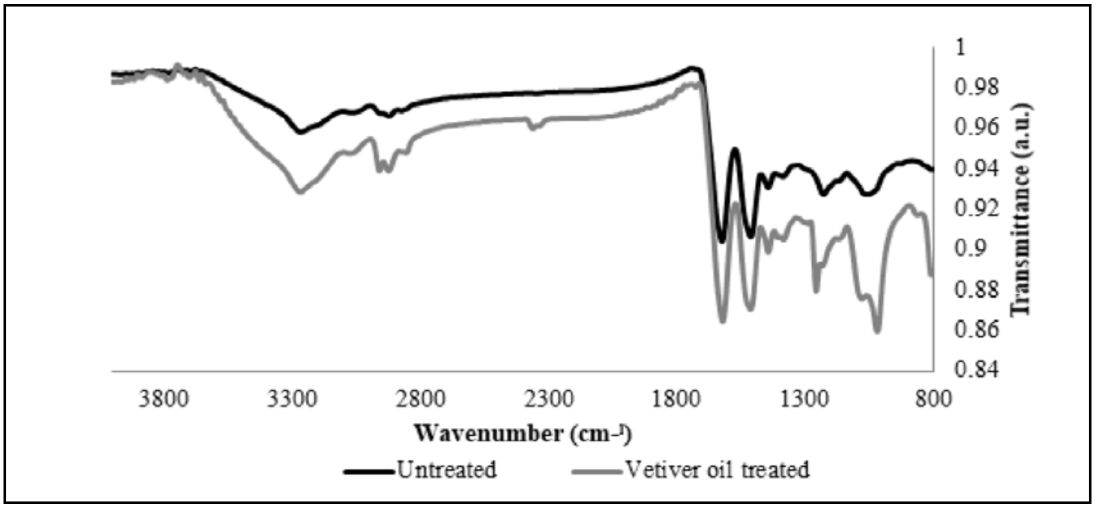

Figure 2. FTIR spectra of wool fabric

microcapsules on the fabric surface and application of other auxiliaries i.e. βeta-cyclodextrin and silicon softener in the treatment bath. Similar finding were reported by Annapoorani *et al.*[2], Bhatt[3], Naikwadi and Sannapapamma[20], and Stan *et al.*[30].

TABLE 1: Effect of aroma treatment on preliminary and mechanical properties of wool fabric

Fabric	Weight g/m²	Count EPI and PPI		Thickness mm	Bending length cm		Flexural rigidity mg-cm	Tensile strength kg		Elongation %	
		Warp	Weft		Warp	Weft	16.29	Warp	Weft	Warp	Weft
Scoured	153.4	55	45	0.4	4.3	2.4	16.39	26.9	20.7	25.9	21.2
Treated	167.8	62	52	0.5	4.7	2.8	22.95	36.2	30.6	35.2	29.7
t-value	5.0*	3.6*	5.0*	0.007	7.4*	6.6*	-	5.2*	4.7*	5.2*	3.0*

*Significant at 5% level of significance.

Table 1 also shows the mechanical properties of the fabrics. Mean bending length and flexural rigidity of treated fabric in warp and weft directions increased as evident from the table. A significant difference ($p<0.05$) was observed in bending length of treated fabric. The results are in line with the findings of Kumari[16], Teli and Chavan[32], and Lim and Setthayanond[18] that the bending length and flexural rigidity values showed slight increase in the magnitude and therefore slight increase in the stiffness of the fabric. The mean tensile strength and elongation of treated fabric also increased as compared to scoured fabric. In aroma treated fabric, a significant change ($p<0.05$) was found in means of tensile strength and elongation values. It may be due to the reduction of projected fibres in wool fabric which made the fabric compact and βeta-cyclodextrin seemed to lessen the restriction of segmental movement of the fibres thereby improving tensile character. These results are in agreement with earlier reports[13, 25].

3.4 Effect of Aroma Treatment on Performance Properties of Wool Fabric

Table 2 depicts performance properties of scoured and aroma treated fabrics. The moisture regain of scoured fabric was 10.49 percent and after aroma treatment it decreased (9.54%). The table further elucidates that the wickability decreased in warp and weft directions of treated fabric in comparison with scoured fabric. The drop test indicated decreased water absorbency as time for absorbance of water drop increased from <51 to <73 seconds after aroma treatment. In aroma treated wool fabric, a significant difference($p<0.05$) was observed in wickability and water absorbency. The decrease in these properties may be due to more surface deposition of microcapsules on the fabric which led to blocking of pores. The results are in line with the studies of Rathinamoorthy and Thilagavathi[22], Singh[26], Janarthanan and Kumar[12] and Yadav[39].

It is apparent from Table 2 that crease recovery angle of scoured fabric decreased from 148 to 141 degree in warp direction and from 88 to 82 degree in weft direction after aroma treatment. Statistically, a non-significant difference ($p>0.05$) was found in crease recovery angle of treated fabric. The results are in agreement with Sukumar and Lakshmikantha[31], Kumari[16] and Naikwadi and Sannapapamma[20] that crease recovery angle decreased as the fabric became stiff due to the deposition of numerous microcapsules on the surface of fabric.

TABLE 2. Effect of aroma treatment on performance properties of wool fabric

Fabric	Moisture regain %	Wickability cm		Water absorbency sec	Crease recovery angle degree		Air permeability CFM
		Warp	Weft		Warp	Weft	
Scoured	10.4	6.2	6.1	<51	148	88	251
Treated	09.5	4.1	4.1	<73	141	82	370
t-value	-	4.1*	4.6*	4.7*	0.04	0.07	6.2*

*Significant at 5% level of significance.

The air permeability of scoured wool fabric increased from 251 to 370 CFM (cubic feet per minute) after aroma treatment as shown in Table 2. A significant change($p<0.05$) was observed in air permeability of treated wool fabric. Thite and Gudiyawar[35] also observed increase in air permeability of fabric after application of microencapsulated essential oils which may be due to the reduction of projected fibres and hairs clinging on the yarn surface allowing more pores.

3.5 Effect of Aroma Treatment on Functional Properties of Wool Fabric

Table 3 shows that the antimoth efficacy of treated fabric was found good in terms of weight loss (7.57%) as compared to scoured fabric (11.83%). Moth mortality was 40 percent in case of treated fabric as compared to 10 percent in case of scoured fabric after 15 days of incubation (Table 3). Udakhe *et al.*[36] also reported that the microcapsule treated samples performed better when tested along with the untreated samples as the moth preferred eating untreated samples than their treated counterpart. Table 3 further reveals that the scoured wool fabric when assessed for antibacterial property in terms of zone of inhibition showed no activity against *Staphylococcus aureus* and *Escherichia coli* while treated fabric exhibited weak antibacterial activity i.e. 02 mm zone of inhibition.

TABLE 3. Effect of aroma treatment on functional properties of wool fabric

Fabric	Moth mortality %	Weight loss %	*Staphylococcus aureus*			*Escherichia coli*			UPF
			Zone of inhibition (mm)	Bacterial count (CFU/ml)	Percent reduction	Zone of inhibition (mm)	Bacterial count (CFU/ml)	Percent reduction	
Scoured	10	11.8	-	31.33×10^8	-	-	35.33×10^8	-	30.25
Treated	40	07.5	02	2.76×10^8	63.45	02	4.08×10^8	61.37	87.07

It was also found that treated fabric had lower bacterial count as compared to scoured fabric for *S. aureus* and *E. coli* with 63.45 and 61.37 percent reduction in bacterial growth, respectively. The results are in line with the findings of Burger *et al.*[7], Naikwadi *et al.*[19], Devprakash *et al.*[8] and Sannapapamma *et al.*[23] that essential oils impart good antibacterial activity in the treated fabric. The scoured wool fabric showed UPF value under very good protection category i.e. 30.25 whereas aroma treated fabric exhibited 87.07 UPF value which came under excellent protection category (Table 3). Due to increased weight and thickness of treated fabric, the pores between the interstices got blocked so this may be a reason for increased UPF value. Geethadevi and Maheshwari[11] investigated the UV protection activity of various essential oil combinations and found that all the essential oil finished fabrics had a good UV protection activity.

3.6 Assessment of Aroma Durability

The durability of aroma treatment on wool fabric in terms of retention and intensity of aroma was assessed against washing, abrasion, ironing and sun-drying.

3.6.1 Retention of Aroma

Fig. 3(a) shows that the aroma was found to be retained even after 30 wash cycles in treated wool fabric as reported by 80 percent respondents. This can be attributed to the fact that wall of microcapsules provided covering to the vetiver essential oil thus protecting it from external environment and also provided a controlled release to give it a longer service life[16]. Aroma was noted to be decreased as the number of abrasion cycles was increased as reported by cent percent respondents that it was retained till 10 abrasion cycles whereas 84 percent respondents agreed that it was retained even after 100 abrasion cycles [Fig. 3(b)]. It is further noticed from Fig. 3(c) that

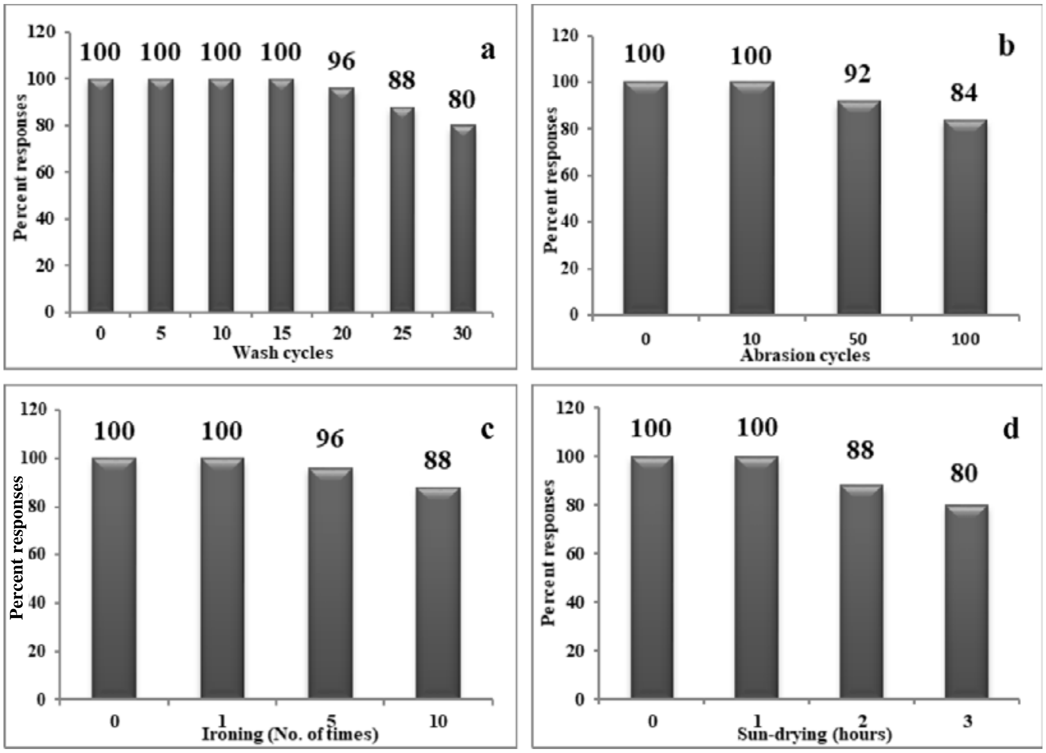

Figure 3. Retention of aroma in treated fabric after (a) washing, (b) abrasion (c), ironing and (d) sun-drying

with increase in number of times of ironing i.e. from 1 to 10 times, retention of aroma decreased but 88 percent respondents agreed that aroma was still retained when treated wool fabric was evaluated after 10 times of ironing. It was found that when the number of hours of sun-drying was increased, retention of aroma was noticed to be decreased. All the respondents (100%) reported that aroma was retained in the treated fabric after 1 hour of sun-drying while 80 percent respondents agreed that aroma was still present in the treated fabric even after 3 hours of sun-drying [Fig. 3(d)]. The results of study are supported by the findings of Geethadevi and Maheshwari[11], Bhatt *et al.*[4], Srivastava and Srivastava[28] and Stan *et al.*[30] that retention of aroma gradually decreased as the exposure to these agencies increased.

3.6.2 Intensity of Aroma

Fig. 4(a) depicts that the intensity of aroma of the treated wool fabric was noted to be very strong after 5 wash cycles (WMS 4.92) then it was perceived strong, moderate, mild and then finally fainted after 10, 15, 20, 25 and 30 wash cycles, respectively. It was noticed that intensity of aroma in treated fabrics decreased as the number of abrasion cycles were increased. Intensity of aroma was felt strong after 10 and 50 abrasion cycles scoring WMS 4.12 and 3.52, respectively then it was regarded as moderate after 100 abrasion cycles (WMS

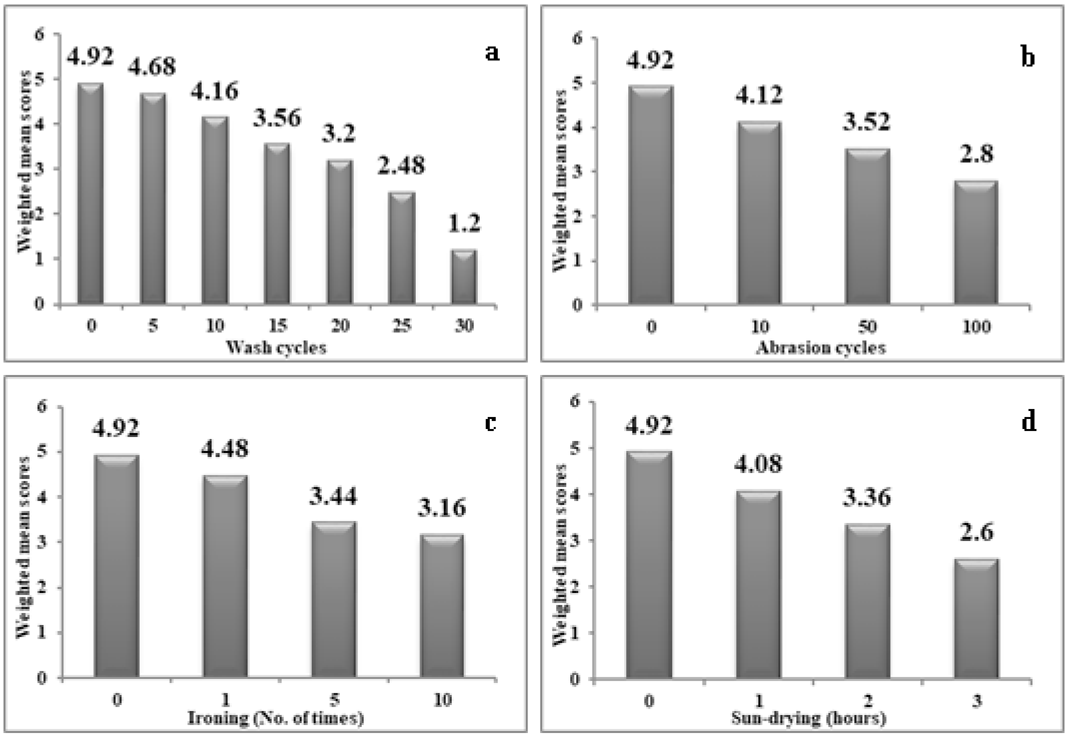

Figure 4. Intensity of aroma in treated fabric after (a) washing, (b) abrasion, (c) ironing and (d) sun-drying

2.80) as observed from [Fig. 4(b)]. Fig. 4(c) shows that intensity of aroma was very strong in the treated sample that was ironed once. Then it was found to be strong and moderate after 5 and 10 times of ironing (WMS 3.44 and 3.16), respectively. It was observed that intensity of aroma got decreased with increased exposure to sunlight. Intensity of aroma was felt as strong in the treated fabric dried for 1 hour (WMS 4.08) in sun and then it was found to be moderate and finally mild when treated wool fabric was dried in sun for 2 and 3 hours (WMS 3.36 and 2.60), respectively [Fig. 4(d)]. The results are found in agreement with Udakhe *et al.* [36], Biswas *et al.*[6] and Bhatt *et al.* [4].

4. CONCLUSION

Wool fabric was treated with microencapsulated vetiver essential oil to impart control release of the fragrance and to assess the effect of aroma treatment on its properties. SEM analysis exhibited presence of numerous microcapsules of spherical shape and uniform size on the surface of treated fabric. FTIR analysis showed presence of various functional groups at different peaks in treated wool fabric. Preliminary and mechanical properties of wool fabric showed an increase after aroma treatment. The air permeability of scoured wool fabric increased after aroma treatment while the moisture properties and crease recovery angle was found to be decreased. Antimoth efficacy of treated fabric was better than scoured wool fabric in terms of weight loss (7.57%) and moth mortality (40%). Aroma treated fabric showed good antibacterial activity against *Staphylococcus aureus and Escherichia coli* with 63.45 and 61.37 percent reduction in bacterial growth, respectively. Scoured wool fabric showed very good UV protection (30.25) but after aroma treatment it provided excellent UV protection (87.07). The aroma durability in terms of retention and intensity of aroma after exposure to different agencies was found very good in treated fabrics. Thus, the microencapsulated vetiver essential oil can be used to impart durable aroma finish onto the wool fabric to develop functionalized textiles having antimoth, antibacterial and UV protection properties without any harmful effect on the physical properties of wool fabric. Vetiver oil also have many reported therapeutic properties therefore, can be used for apparel, home, medical and healthcare textiles that could be beneficial for the wearer/user. The wool fabric being highly hygroscopic in nature absorbed more aroma and also retained it for a longer time hence, considered very suitable fabric for development of aroma textile.

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