World Academy of Science, Engineering and Technology
International Journal of Materials and Textile Engineering
Vol:12, No:2, 2018

Antimicrobial and Aroma Finishing of Organic Cotton Knits Using Vetiver Oil Microcapsules for Health Care Textiles

K. J. Sannapapamma, H. Malligawad Lokanath, Sakeena Naikwadi

Abstract**—**Eco-friendly textiles are gaining importance among the consumers and textile manufacturers in the healthcare sector due to increased environmental pollution which leads to several health and environmental hazards. Hence, the research was designed to cultivate and develop the organic cotton knit, to prepare and characterize the Vetiver oil microcapsules for textile finishing and to access the wash durability of finished knits. The cotton *SAHANA* variety grown under organic production systems was processed and spun into 30 single yarn dyed with four natural colorants (Arecanut slurry, Eucalyptus leaves, Pomegranate rind and Indigo) and eco dyed yarn was further used for development of single jersy knitted fabric. *Vetiveria zizanioides* is an aromatic grass which is being traditionally used in medicine and perfumery. Vetiver essential oil was used for preparation of microcapsules by interfacial polymerization technique subjected to Gas Chromatography Mass Spectrometry (GCMS), Fourier Transform Infrared Spectroscopy (FTIR), Thermo Gravimetric Analyzer (TGA) and Scanning Electron Microscope (SEM) for characterization of microcapsules. The knitted fabric was finished with vetiver oil microcapsules by exhaust and pad dry cure methods. The finished organic knit was assessed for laundering on antimicrobial efficiency and aroma intensity. GCMS spectral analysis showed that, diethyl phthalate (28%) was the major compound found in vetiver oil followed by isoaromadendrene epoxide (7.72%), beta-vetivenene (6.92%), solavetivone (5.58%), aromadenderene, azulene and khusimol. Bioassay explained that, the vetiver oil and diluted vetiver oil possessed greater zone of inhibition against *S. aureus* and *E. coli* than the coconut oil. FTRI spectra of vetiver oil and microcapsules possessed similar peaks viz., C-H, C=C & C=O stretching and additionally oil microcapsules possessed the peak of 3331.24 cm^{-1} at 91.14 transmittance was attributed to N-H stretches. TGA of oil microcapsules revealed that, there was a minimum weight loss (5.835%) recorded at 467.09°C compared to vetiver oil i.e., -3.026% at the temperature of 396.24°C. The shape of the microcapsules was regular and round, some were spherical in shape and few were rounded by small aggregates. Irrespective of methods of application, organic cotton knits finished with microcapsules by pad dry cure method showed maximum zone of inhibition compared to knits finished by exhaust method against *S. aureus* and *E. coli*. The antimicrobial activity of the finished samples was subjected to multiple washing which indicated that knits finished with pad dry cure method showed a zone of inhibition even after 20^{th} wash and better aroma retention compared to knits finished with the exhaust method of application. Further, the group of respondents rated that the 5^{th} washed samples had the greater aroma intensity in both the methods than the other samples. Thus, the vetiver microencapsulated organic cotton knits are free from hazardous chemicals and have multi-functional properties that can be suitable for medical and healthcare textiles.

Keywords**—**Exhaust and pad dry cure finishing, interfacial polymerization, organic cotton knits, vetiver oil microcapsules.

K. J. Sannapapamma is with the University of Agricultural Sciences, Dharwad, India (e-mail: sannapapammakj@uasd.in).

I. Introduction

THE textile industry in the medical sector is developing at an incredible rate with achievements in the areas *viz.*, infection control, barrier material, wound care products, hospital clothing and bedding due to innovative application in today's healthcare environment. The main requirement of the medical textile material is biodegradability and biocompatibility [1]. Many textile manufacturers and research laboratories are currently engaged in developing various eco-friendly finishes for textile products. The textile finish needs to have properties such as skin friendly, environment friendly and wash resistance for its effective usage for a longer period. Antimicrobial textiles with improved functionality find a variety of applications such as health and hygiene products, specially the garments worn close to the skin and several medical applications, such as infection control and barrier material. Antimicrobial fabrics are important not only in medical applications but also in terms of daily life usage [2].

Recently, a demand for natural fiber-based textile coloured and finished with various bio-resources is gaining academic, research and industrial importance because of the increased global awareness of environmental pollution. Therefore, wet processing of textile carried out by using various plant extracts is gaining greater attention due to the presence of the inherent colouring compound and functional properties. These textiles exhibited excellent antimicrobial, UV protective and aroma functions. However, organic cotton has been gradually gaining popularity in modern times, since people all over the world are becoming more aware of global warming, health, pollution and environmental protection issues. Hence, greater numbers of the farming community and spinning mills are opting for organic cotton production which is being used in knitting and weaving mills. Thus, the demand for organic cotton is increasing day by day, as the awareness among consumers is growing for the eco-friendliness from the fibre stage itself.

Nature is providing all the resources to mankind for their sustenance. Natural dyes, pigments, aromatic sources and many more are the best examples which are being used in the textile wet processing since time immemorial. *Vetiveria zizanioides* is an aromatic grass and one of the natural resources that has great application for the well-being of humans. It is popularly known as Khas Khas, Khas or Khus grass in India and is the common source of well-known oil of vetiver, which is being used in medicine and perfumery. A major portion of the oil consists of sesquiterpenoids, hydrocarbon and their oxygenated derivatives. Phyto-chemical screening of the powdered leaves exhibited presences of alkaloids, falvonoides, tannins, phenols, terpenoids and saponins. Roots are highly aromatic, antimicrobial, antifungal, UV blocking, mosquito repellent, cooling, haemostatic, expectorant, insomnia, skin diseases and anti-oxidant [3].

Essential oils are a rich source of biological active compounds but their application is limited because of the volatile nature and chemical instablility in the presence of oxygen, moisture, heat and repeated laundering. The stability of the essential oil can be enhanced by micro encapsulation by different techniques. Chemically microencapsulation methods are based on polymerisation or poly condensation mechanisms that may be carried out through different techniques and found to be appropriate for textile application and proved that the loaded microcapsules retained their efficiency even after 35 wash cycles [4] and then begins to decrease gradually [5]. The microcapsules, prepared by interfacial polymerization, have several advantages over others, which include high yield encapsulation, inexpensive preparation cost and ease of regulation of the encapsulation process [6]. The *vetiver* essential oil has been traditionally used in aroma therapy for relieving stress, anxiety, nervousness, tension and insomnia for a long time. It has a multi-functional value which can be used as an eco-friendly finishing agent on textiles to impart better functional properties. Hence, the oil was purposively selected for preparation of microcapsules using Cetyltrimethyle bromide as shell material through interfacial polymerization technique with following objectives; to develop organic cotton knits, characterization of vetiver oil microcapsules, and finishing and wash durability of vetiver oil microencapsulated organic cotton knits.

II. Material and Methods

A. Development of Organic Cotton Knits

Organic cotton *SAHANA* variety grown under organic production systems was processed and spun into 30 count single yarn (Table I). The spun yarn was subjected to natural dyeing with four natural colourants *viz*., Eucalyptus, Areca nut slurry, Pomegranate rind and Indigo. The eco-dyed yarns were used for development of single jersy knitted fabric (24 stitch density, 0.58 mm thickness and busting strength of 45/p).

B. Preparation and Characterization of Vetiver Essential Oil Microcapsules

1. Bio-Assay Test

Vetiver essential oil (*Vetiveria zizanioidias*) was subjected to bio assay test to determine the antimicrobial efficiency of the oil against the test organism *Staphylococcus aureus and Escherichia coli*. The paper disc method was adopted for diffusion of the essential oil to determine the antimicrobial activity.

TABLE I
FIBRE AND YARN PHYSICAL PROPERTIES OF ORGANIC COTTON GROWN UNDER ORGANIC PRODUCTION SYSTEM

Sl No	Parameter	Unit
I	**Fibre properties (*SAHANA* Variety cotton)**	
1	2.5 % SL (mm)	26.52
2	U.R. (%)	47.42
3	Mic.(g/in)	3.40
4	Tenacity (g/tex)	20.18
5	Elongation (%)	5.70
II	**Yarn evenness properties (1000mtrs)**	
1	Thin Places (-50%)	1330
2	Thick Places (+50)	2056
3	Neps (+200)	2714
4	Total Imperfection	6100
5	Uniformity Ratio	19.93
6	CSP (Count strength Product)	1797.03
III.	**Yarn physical properties**	
1	Twist per inch (TPI)	24.30
2	Tenacity (g/tex)	10.90
3	Breaking elongation (%)	6.20

2. Microencapsulation Technique

Microencapsulation process involves three different steps i.e. formation of the wall around the core material; protection of the core material and preventing the entry of undesirable material, and finally, the release of core material by a control release rate [7].

Based on the literature, three microencapsulation methods, namely, microcapsules with melamine formaldehyde, in-situ polymerization and interfacial polymerization were initially tried to get the microcapsules of vetiver essential oil. Among the methods, the effective capsules were found in the interfacial polymerization technique. Hence, this technique was adopted for final preparation of microcapsules.

3. Preparation of Vetiver Oil Microcapsules

Interfacial polymerization technique was used for preparation of microcapsules using the following recipe: 5 ml of Cetyltrimethyle bromide (CTAB) dissolved in 95 ml of deionised water and stirred at 500 rpm for 15 min. A diluted vetiver oil (Vetiver oil: 2 ml, Coconut oil: 4 ml) and Dicyclohexyl methane 4,4 dilsocyanate (3 ml) solution was mixed properly and added into the Tween 20 solution. After 5 min, ethylene diamine (5 ml) was dissolved in water (5 ml) then added in to the core solution and the temperature of the solution was maintained at 40°C. The solution was stirred at 600-800 rpm for 15-20 minutes. The prepared solution was subjected to filtration using Whatman No. 1 filter paper. The filtered microcapsules stock was dried under shade and stored in a refrigerator for further application.

4. Characterization of Essential Oil Microcapsules

The powdered essential oil microcapsules were characterized under SEM, TGA and FTIR to know the size of the capsules, functional groups and thermal property.

5. Fourier Transform Infrared Spectroscopy

FTIR is a technique which is used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas.

The surface functional group of essential oil microcapsules was examined using Perkins Elmer FT-IR spectrometer CC99589 spectrum two.

6. TGA

TGA is a method of thermal analysis where change in physicochemical properties of materials is measured as a function of increasing temperature. The thermal stability of the polymer and microcapsules was evaluated with a Mettler Model TGA/SDTA851 TGA. The samples of 5 mg each were heated to 600°C at a rate of 10°C/min under nitrogen atmosphere.

7. SEM

Vetiver essential oil microcapsules were analyzed under SEM *JEOL JSM* 5400 to find out the formation, size of microcapsules and presence of the microcapsules on surface of the treated organic knits.

C. Antimicrobial Efficiency and Wash Durability of Vetiver Oil Microencapsulated Organic Cotton Knits

A specially designed multi striped organic cotton knitted fabric was finished with oil encapsulated with CTAB through exhaust and pad dry cure method. Antimicrobial activity of the treated fabrics was assessed by AATCC 147 test method to determine the antimicrobial efficiency and to estimate the degree of antimicrobial activity after multiple washes.

D. Olfactory Analysis

Treated organic cotton knits were subjected to olfactory analysis to rate the fragrance retention after washing cycles by 30 panel members. Sensory evaluation was carried out by administering a self-structured questionnaire to elicit the olfactory analysis for treated fabrics after every wash cycle (5-20th). The experimental data were analysed by using Weighted Mean Score (WMS), frequencies, percentage and standard deviation.

III. Results and Discussion

A. Characterization of Vetiver Essential Oil and Oil Microcapsules

1. GCMS Spectral Analysis of Vetiver Essential Oil

The major separated compounds of vetiver oil are identified by using NIST library search method. The chemical compound of vetiver oil is illustrated in Table II, with retention time, area percentage, base peak, as well as the chemical structure and its formula. Based on the percentage composition, retention time and base peak, the percent composition of diethyl phthalate was found to be greater at the retention time of 13.588 with base m/z of 149.05 followed by isoaromadendrene epoxide (7.72%) at retention time 16.36 with base peak of 121.10, beta-vetivenene (6.92%) at retention time 12.210 with base m/z of 145.10, solavetivone (5.58%) at retention time 23.402 with base m/z 136.10, respectively. Further, alpho-cadina 4-9 dienenaphthalane (4.80%) was found at retention time of 9.477 with base m/z peak 105.05 followed by aromadendrene (4.63%) and viridiflorol (4.32%), at retention time of 15.06 with base peak of 81.05 m/z. However, percentage area of other compounds present in the source ranged between 2% to 3% namely, azulene (2.62), 2-Propenal (2.59), octahydro dimethyl (2.41), Acetate (2.42), 2-Propenol (2.05), cyclopropazulene (2.01). The area percentage of the compounds ranged between 1% and 2% namely, cyclopropene azulene (1.97), naphthalene (1.87), cyclohexen (1.52) khusimol (1.38), benzene (1.32), gaama-murolene (1.30%), azulene (1.25%), valerenyl acetate (1.20%), isopropyle (1.17%) and alpha-eudesma (1.16%) at different R time retention with varied base peak. Among the major compounds, the greater percentage of area was attributed by Diethyl phthalate which is a colourless liquid with slight aromatic odour, low volatability and widely used as a plasticizer in a wide variety of consumer products including plastic packaging films, cosmetic formation and toiletries as well as in medical treatment stated by World Health Organization. However, aromadenderene was the second most major compound and it has a sesquiterpenoid hydrocarbon exhibited antimicrobial property [8]. Further, beta-vetivenene, solavetivone, aromadenderene and alpha cadina naphthalene, viridiflorol, azulene and khusimol showed strong antioxidant activity. Several studies showed that, vetiver oil possessed antibacterial activity against various bacterial strains like *Staphylococcus aureus, Streptococcus pyogenes, Escherichia coli* and *Corynebacterium antioxidenet* activity [9].

2. Antimicrobial Efficiency of Pure Vetiver Oil, Diluted Vetiver Oil and Coconut Oil against Test Organisms through Bio-Assay Method

The pure vetiver oil, coconut oil and diluted vetiver oil, assessed for antimicrobial activity through bioassay method (Paper disc diffusion), showed that the vetiver oil (8.80±0.18, 7.68±0.45) possessed greater zone of inhibition against *S. aureus* and *E. coli* than the diluted vetiver oil (8.20±0.17, 7.00±0.18) due to the presence of a large quantity phyto constituents and numerous bio active agents, as discussed in Table II. However, the coconut oil did not exhibit an inhibition zone, as shown in the Figs. 1 and 2. Similar results were found in the study "Microencapsulation of essential oil by interfacial polymerization using polyurea as a wall material" which stated that oregano and sage essential oil were tested as an antimicrobial and antifungal property and presented an excellent reduction at the bacteria and fungi test [10].

3. FTIR Spectra of Vetiver Oil

From Fig. 3, the two strong peaks found between the region 2923.40 cm^{-1} to 2854.02 cm^{-1} exhibited the presence of the functional group assigned to C-H stretching. Whereas, the peaks found in the region 1743.72 cm^{-1} were assigned with very strong amides and carboxylic acids with C=O functional group. The peaks ranged between 1458.21 cm^{-1} to 1377.19 cm^{-1} showing the movement of amine and amide group with NH_2 bending. In the spectrum, it can be observed that the medium size peaks at 1156.78 cm^{-1} and 1111.84 cm^{-1} can be attributed to C-O stretches. However, 889.24 cm^{-1} and 721.77 cm^{-1} peaks can be attributed to the phenyl group *i.e.,* C-H and C-C, which is responsible for the aromatic and antimicrobial properties.

TABLE II
GCMS SPECTRAL ANALYSIS OF VETIVER ESSENTIAL OIL

Sl No	R. Time	Area %	Base peak m/z	Name	Formula
1	8.836	2.41	134.15	Naphthalene, 1,2,3,5,6,7,8,8a-Octahydro-1,8a-Dimethyl-7- (1-Methylethenyl)	$C_{15}H_{24}$
2	9.477	4.80	105.05	6.Alpha.-Cadina-4,9-Diene, (-)- $$ Naphthalene, 1,2,4a,5,6,8a-Hexahydro-4,7-Dimethyl-1- (1-Methylethyl)	$C_{15}H_{24}$
3	9.602	1.32	160.10	Benzene, 1,3,5-Trimethyl-2- (1-Methylethenyl)-Styrene, .Alpha.,2,4,6-Tetramethyl- $$ 2-Isopropenyl-1,3,5-Tr	$C_{12}H_{16}$
4	9.732	2.62	161.15	Patchoulene/ AZULENE, 1,2,3,3A,4,5,6,7-OCTAHYDRO-1,4-DIMETHYL-7- (1-METHYLETHENYL)-, [1R- (1.ALPHA.,3A.BETA.,4.ALPHA.,7.BETA.)]-	$C_{15}H_{24}$
6	9.944	1.30	161.10	Gamma.-Muurolene Naphthalene, 1,2,3,4,4a,5,6,8a-Octahydro-7-Methyl-4-Methylene-1- (1-Methylethyl)	$C_{15}H_{24}$
7	10.289	2.01	161.10	1h-Cycloprop[E]Azulene, Decahydro-1,1,7-Trimethyl-4-Methylene-, [1ar-(1a.Alpha.,4a.Alpha.,7.Alpha.,7a.B	$C_{15}H_{24}$
8	10.732	1.25	119.10	Beta.-Guaiene Azulene, 1,2,3,4,5,6,7,8-Octahydro-1,4-Dimethyl-7- (1-Methylethylidene)	$C_{15}H_{24}$
9	10.927	1.97	161.15	1h-Cycloprop[E]Azulene, 1a,2,3,4,4a,5,6,7b-Octahydro-1,1,4,7-Tetramethyl-, [1ar-(1a.Alpha.,4.Alpha.,4a.Beta.,7b.Alpha	$C_{15}H_{24}$
10	11.151	1.20	145.10	(Z)-Valerenyl Acetate Z-Valerenyl Acetate	$C_{17}H_{26}O_2$
11	12.210	6.92	145.10	Beta.-Vetivenene	$C_{15}H_{24}$
12	12.480	1.29	131.10	Gamma.-Himachalene	$C_{15}H_{24}$
13	12.608	1.38	149.15	Rosifoliol	$C_{15}H_{24}O$
14	13.398	1.52	187.10	2-Cyclohexen-1-Ol, 1,2,4,4-Tetramethyl-3- (3-Methyl-1,3-Butadienyl)-, (E)- (.+-.)- $$ (E)-1,2,4,4-Tetramethyl-3- (3'-Me	$C_{15}H_{24}O$
15	13.588	28.00	149.05	Diethyl Phthalate $$ 1,2-Benzenedicarboxylic Acid, Diethyl Ester $$ Phthalic Acid	$C_{12}H_{14}O_4$
16	13.795	1.17	119.10	8-Isopropyl-1,3-Dimethyltricyclo[4.4.0.0~2,7~]Dec-3-Ene $$ Alpha-Copaen	$C_{15}H_{24}$
17	15.065	4.32	81.05	Viridiflorol	$C_{15}H_{26}O$
18	16.362	7.72	121.10	Isoaromadendrene Epoxide	$C_{15}H_{24}O$
19	16.883	2.42	202.10	Acetate, [6- (Acetyloxy)-5,5,8a-Trimethyl-2-Methyleneperhydro-1-Naphthalenyl]Methyl Ester	$C_{19}H_{30}O_4$
20	16.980	1.84	105.05	Naphthalene, Decahydro-4a-Methyl-1-Methylene-7- (1-Methylethenyl)-, [4ar-(4a.Alpha.,7.Alpha.,8a.Beta.)]- $	$C_{15}H_{24}$
21	18.786	1.16	189.10	Alpha.-Eudesma-4,6-Diene	$C_{15}H_{24}$
22	23.241	2.05	105.05	2-Propenal, 3- (2,4,5,6,7,7a-Hexahydro-3	$C_{15}H_{22}O$
23	23.402	5.58	136.10	Solavetivone $$ 2-Isopropenyl-6,10-Dimethylspiro	$C_{15}H_{22}O$
24	23.944	2.59	105.05	2-Propenal, 3- (2,4,5,6,7,7a-Hexahydro-3	$C_{15}H_{22}O$

TABLE III
ANTIMICROBIAL EFFICIENCY OF PURE VETIVER OIL, DILUTED VETIVER OIL AND COCONUT OIL AGAINST TEST ORGANISMS THROUGH BIO-ASSAY METHOD

Organism	Vetiver oil	Diluted vetiver oil (1:2)	Coconut oil
Staphylococcus aureus	8.80 ± 0.18	8.20±0.17	NI*
Escherichia coli	7.68 ± 0.45	7.00±0.18	NI*

Figures in parentheses indicate Mean, Note: NI- No inhibition

Fig. 1 Antimicrobial efficiency of pure vetiver oil, diluted vetiver oil and coconut oil against *S. aureus* through bio-assay method (Paper disc diffusion method)

Fig. 2 Antimicrobial efficiency of pure vetiver oil, diluted vetiver oil and coconut oil against *E. coli* through bio-assay method

4. FTIR Spectra of Vetiver Oil Microcapsules

FTIR spectra of vetiver microcapsules possessed several functional groups (Fig. 4). The peak of 3331.24 cm^{-1} at 91.14 transmittance percentage is attributed to N-H stretches. Two important peaks were observed between the range 2922.81 cm^{-1} and 2853.11 cm^{-1}, is assigned to strong C-H stretching. This stretching is attributed to the reaction with the major compound of vetiver oil i.e., Diethyle phthylate ($C_{12}H_{24}O$) reacts with dicyclohexylmethane and ethylene diamine to form strong C-H_2 bonds. A medium intensity alkenes group was found at 2262.70 cm^{-1} with C≡C stretching. The functional group of ketones, aldehyde, easters, carboxylic acid, amide, alkenes and benzenes exhibited in the region of 1745.01 cm^{-1} is assigned to C-O stretching followed by 1629.90 cm^{-1} and 1555.39 cm^{-1} assigned to NH_2 stretching. Further, remaining peaks ranged between 898.52 cm^{-1} to 651.37 cm^{-1} which can be attributed to an aromatic and phenyl group due to presence of compound namely, diethyl phthalate, isoaromadendrene epoxide, beta-vetivenene, solavetivone, aromadenderene and alpha cadina naphthalene, viridiflorol, azulene and khusimol found in vetiver oil.

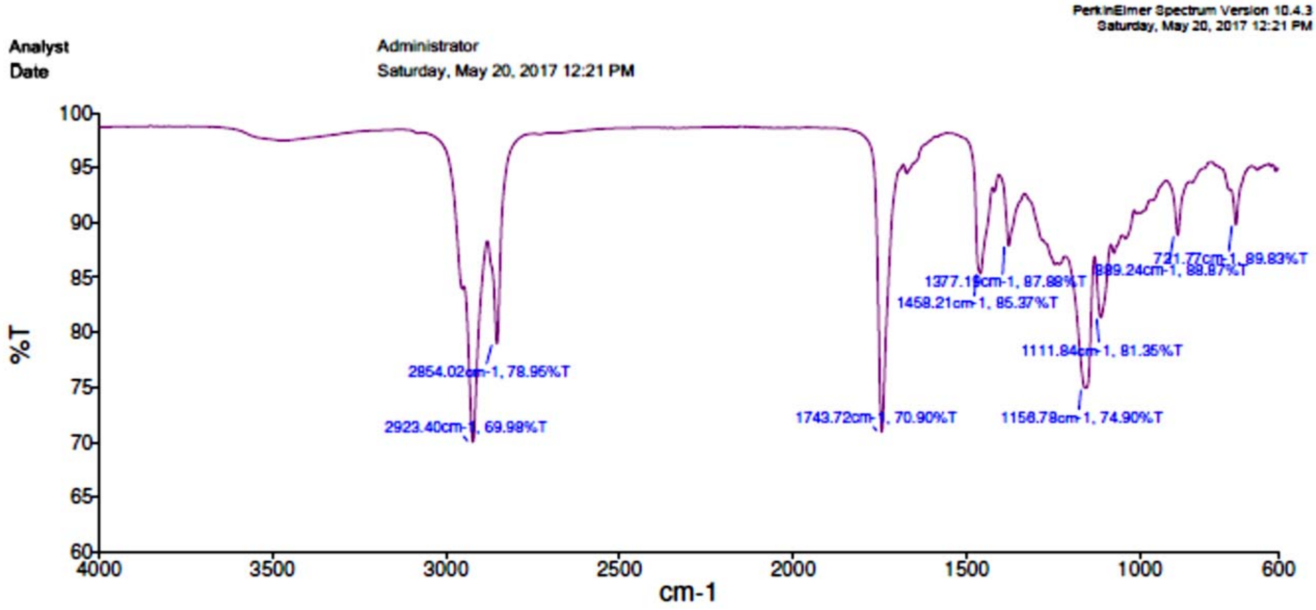


Fig. 3 FT-IR spectra of vetiver oil

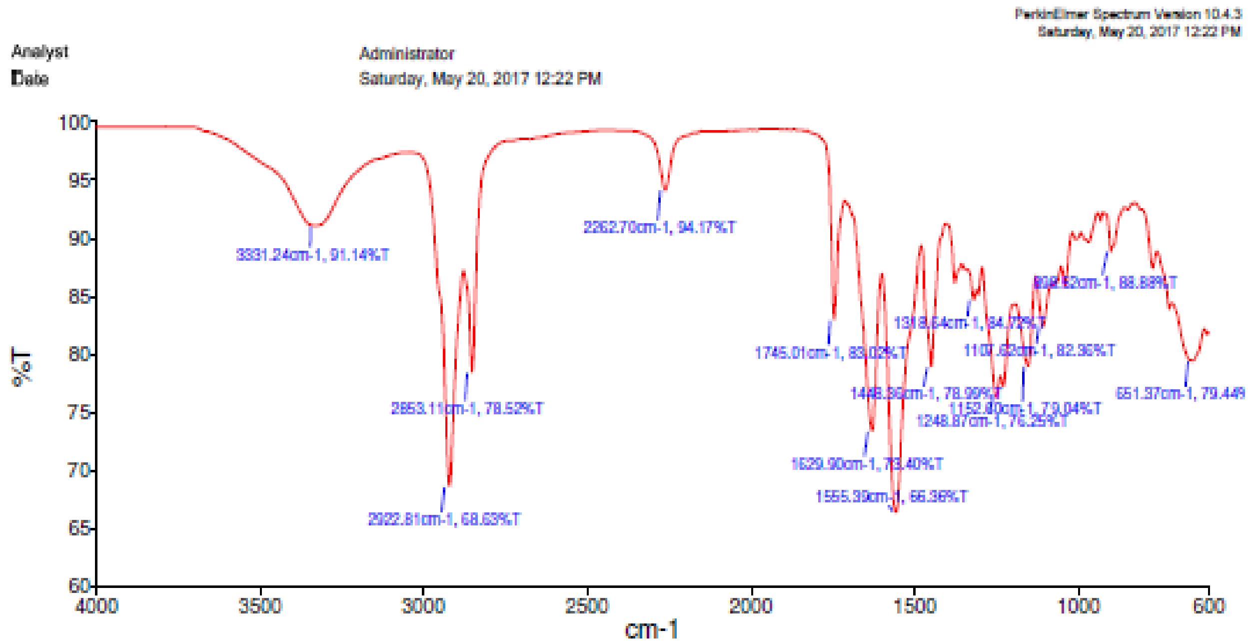


Fig. 4 FT-IR spectra of vetiver oil microcapsules

5. FT-IR Spectra of Combined Vetiver Oil and Oil Microcapsules

Fig. 5 showed that the combined spectra of vetiver oil and oil microcapsules exhibited three major peaks present in oil also appeared in the oil microcapsules namely, two strong peaks were observed between the range of 2923.40 cm^{-1} to 2854.02 cm^{-1} assigned to strong C-H stretching at 69.98 and 78.95 transmittance percentage followed by 1743.72 cm^{-1} with transmittance percentage of 70.90 is assigned to C=O stretching indicated that, the major functional groups present in oil were also present in the oil microcapsules with reduced intensity. This may be due to the active presence of shell material used for the preparation of the vetiver oil microcapsules.

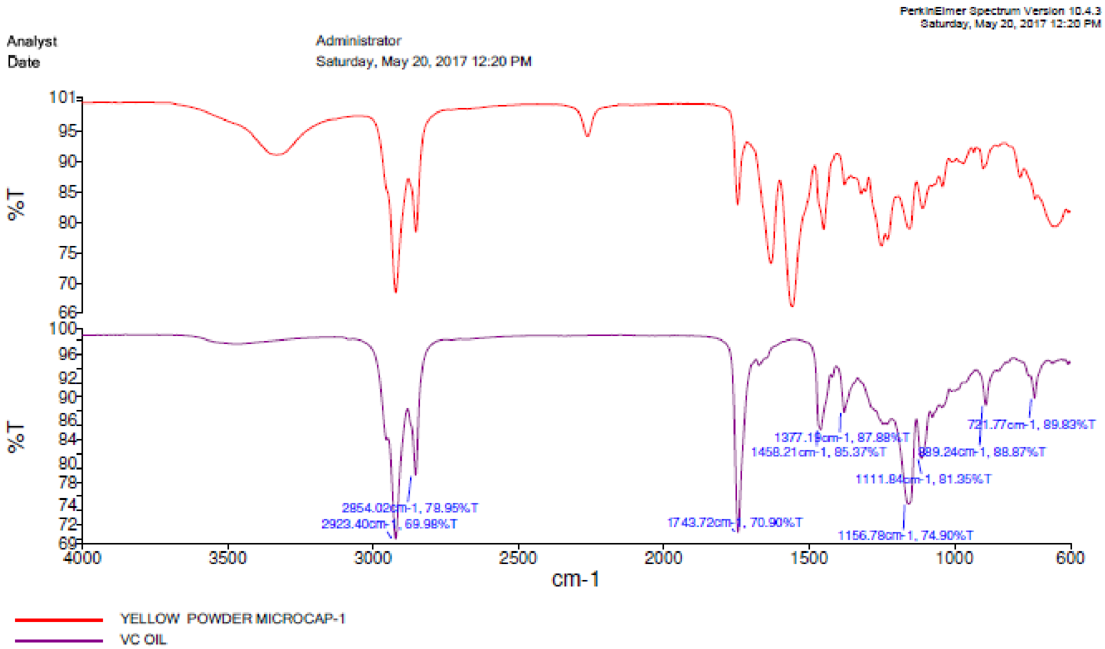


Fig. 5 FT-IR combined spectra of vetiver oil and vetiver oil microcapsules

6. TGA of Vetiver Oil and Oil Microcapsules

Diluted vetiver oil (vetiver oil + coconut oil 1:2) and vetiver microcapsules were subjected to TGA showed that substantial weight loss was observed in both the materials subjected to different temperature intervals.

The weight loss of oil was started from 108. 92°C temperature with the weight of 98.60%, whereas 59.22% of weight was attained at 265.03°C. Further, the complete loss of oil weight was observed at 398.24°C (-398.24%).

The TGA of oil microcapsules analyzed and recorded that, the weight loss was initiated at 135.16°C with weight of 97.08%, and at the temperature of 327.23°C, the oil microcapsules attained the weight of 43.40% and complete discharge of weight (5.835%) was observed at 467.09°C.

In general, the TGA of vetiver oil microcapsules was found to be better as compared to the TGA of vetiver oil. This may be due to combined effect of core material (vetiver oil) and wall material (dicyclohexylmethane) used during microencapsulation which helps to retain and improve the weight of core materials compared to vetiver oil (Figs. 6 and 7).

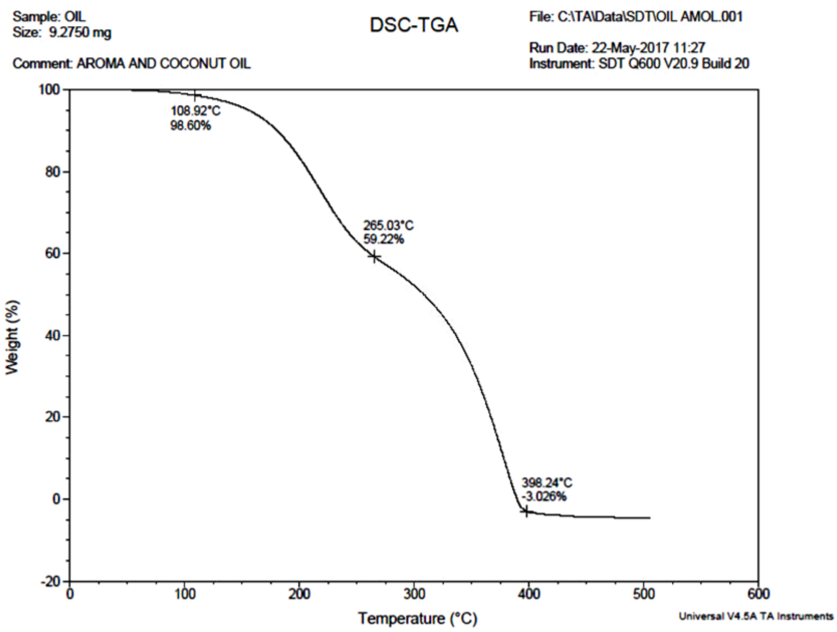


Fig. 6 TGA of vetiver oil

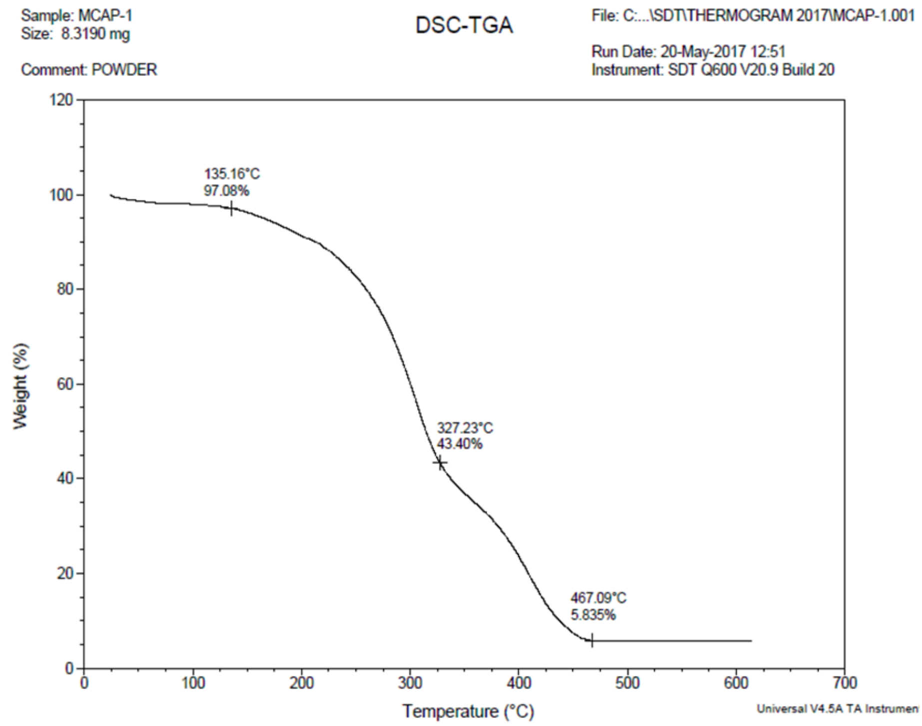


Fig. 7 TGA of vetiver oil microcapsules

7. Size Distribution of Vetiver Microcapsules

The prepared microcapsules were subjected to SEM to determine the size of the distribution of vetiver microcapsules. The size of the vetiver microcapsules ranged from 1.73 µ to 60.02 µ and was categorized into small size (20.85±6.30), medium size (40.29±13.96), and big size (60.02±14.16) and aggregated form 1.73±0.423. Table IV and Fig. 8 reported that, the morphology of microcapsules were fairly irregular. This may be due to the influence of preparation conditions, core and shell material concentration and liquor ratio [10].

TABLE IV
SIZE DISTRIBUTION OF VETIVER ESSENTIAL OIL MICROCAPSULES

Capsule size category	Vetiver oil microcapsules
Small size	20.85 ± 6.30
Medium size	40.29 ± 13.96
Big size	60.02 ± 14.16
Aggregated form	1.73 ± 0.423

Figures in parentheses indicate Mean ± Standard deviation

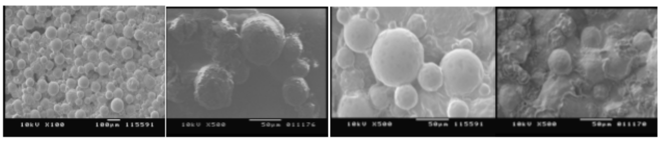


Fig. 8 SEM images of vetiver oil microcapsules

B. Antimicrobial Efficiency of Vetiver Finished Organic Cotton Knits and Wash Durability

Antibacterial efficiency of vetiver finished samples against gram positive organism *Staphylococcus aureus* and negative organism *Escherichia coli* is reported below.

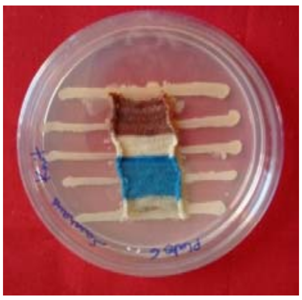
Irrespective of source and methods of application, all the treated samples exhibited maximum antibacterial efficiency against gram positive organism *Staphylococcus aureus* than gram negative organism *Escherichia coli*. This may be due to the gram positive bacteria being more susceptible to natural herbal plants extracts compared to gram negative bacteria [11]. The fabric treated with geranium extract showed a higher zone of inhibition against *S. aureus* compared to *E. coli*, as *S. aureus* is more sensitive to geranium extract compared to *E. coli* [12].

The vetiver oil microcapsules treated knits by pad dry cure method showed maximum antimicrobial efficiency against gram positive organism *S.aureus* and gram negative organism *E. coli* with zone of inhibition 9.2 ± 0.570 and 8.5 ± 0.570 compared to the knits treated with vetiver oil microcapsules through exhaust method against *S.aureus* and *E.coli* with zone of inhibition 7.4 ± 0.418 and 6.5 ± 0.790, respectively. This may be due to the absorption and deep deposition of vetiver oil microcapsules on the surface of the fabric by the pressure of rollers during finishing process (Table V and Fig. 9). The results are on par with a study on "Antibacterial finish for cotton fabric from herbal products" that reported microencapsulated fabric is not significantly affected due to repeated laundering when compared with that of direct treated fabrics. This perhaps is due to the sustained release of antibacterial compounds over repeated laundering from herbal products because of the microencapsulation technique and also the use of resin (cross linking agent) during the finishing process, even though citric acid was used in the direct method [13], [14]. Whereas in the case of exhaust method, the microcapsules are distributed on the surface without firm bonding, which resulted in fair antibacterial durability.

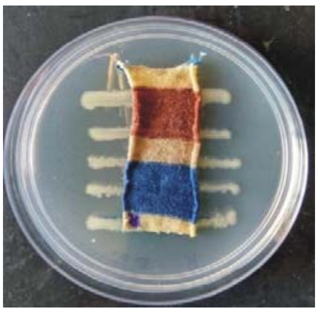
TABLE V
ANTIMICROBIAL EFFICIENCY OF VETIVER FINISHED ORGANIC COTTON KNITS AND WASH DURABILITY

	Method of finishing	Organisms	Wash cycles	Control	Vetiver oil microcapsules
Zone of inhibition (mm)	Exhaust method	*S. aureus*	0	NI	**7.4 ± 0.418**
			5		6.3 ± 0.83
			10		4.3 ± 0.570
			15		1.8 ± 0.570
			20		1.0 ± 0.5
		E. coli	0	NI	**6.5 ± 0.790**
			5		4.00 ± 0.5
			10		1.9 ± 0.418
			15		0.9 ± 0.41
			20		0.7 ± 0.273
	Pad dry cure method	*S. aureus*	0	NI	**9.2 ± 0.570**
			5		8.4 ± 0.547
			10		6.9 ± 0.418
			15		5.0 ± 0. 790
			20		2.1 ± 0.41
		E. coli	0	NI	**8.5 ± 0.50**
			5		6.4 ± 0.82
			10		5.4 ± 0.41
			15		2.0 ± 0.5
			20		1.1 ± 0.65

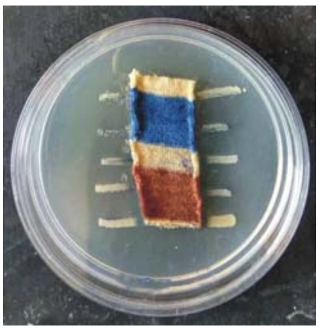
After subjecting to the set of washing cycles, all the treated fabrics possessed decreased antibacterial efficiency compared to unwashed samples. However, greater durability of antibacterial efficiency was found in the knits treated with vetiver oil microcapsules through pad dry cure method, which showed zone of inhibition even after 15 washes. The above results are supported by the SEM images of samples treated with microcapsules finished by exhaust method and pad dry cure method with washing intervals (Figs. 10 and 11).



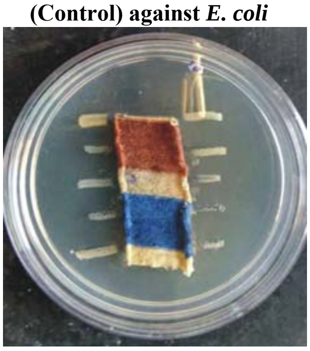
Organic cotton knits (Control) against *S. aureus*



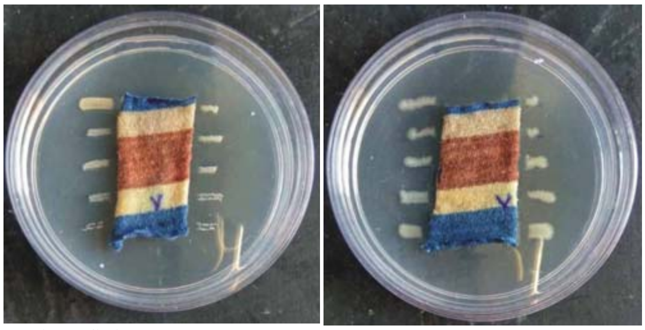
Organic cotton knits (Control) against *E. coli*



Vetiver finished sample (Exhaust) against *S. aureus*

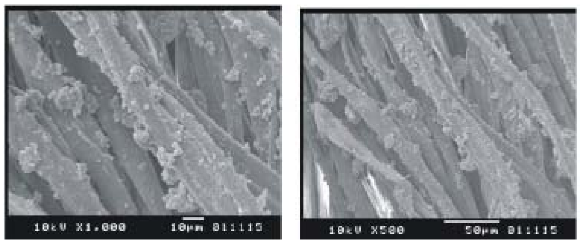


Vetiver finished sample (Exhaust) against *E. coli*

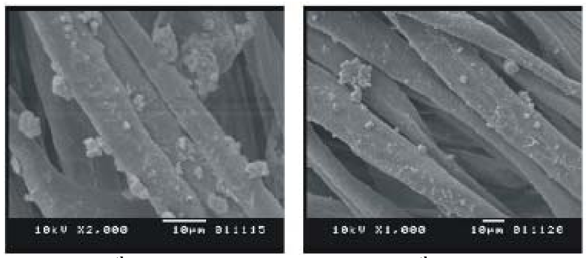


Vetivar finished sample (Pad dry cure) against *S. aureus* **Vetivar finished sample (Pad dry cure) against *E. coli***

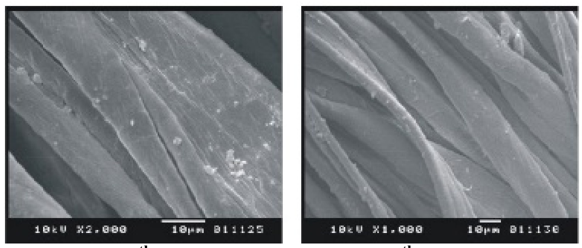
Fig. 9 Antimicrobial efficiency of Vetiver oil microcapsules finished organic cotton knits against microorganisms



Microencapsulated organic knits (control)

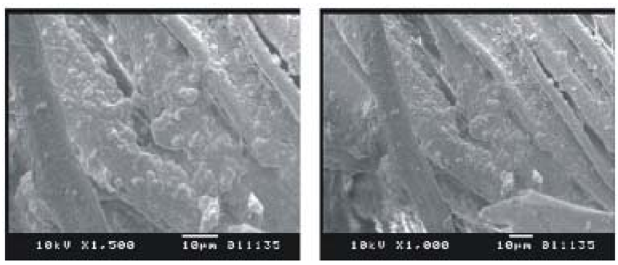


5th wash **10th wash**

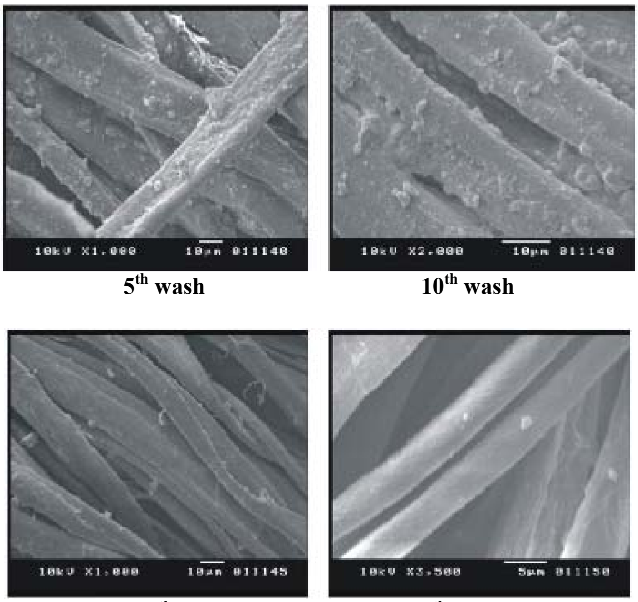


15th wash **20th wash**

Fig. 10 Surface morphology (SEM) of organic cotton knits finished with vetiver oil microcapsules by exhaust method and wash durability



Microencapsulated organic knits (control)



5th wash **10th wash**

15th wash **20th wash**

Fig. 11 Surface morphology (SEM) of organic cotton knits finished with vetiver oil microcapsules by pad dry cure method and wash durability

C. Olfactory Analysis of Vetiver Oil Microcapsules Finished Organic Cotton Knits

The treated and washed organic cotton knits were evaluated on the basis of olfactory analysis against aroma intensity by a panel of 30 experts (15- teachers and 15- students). Based on the ratings given by the experts, the WMS was calculated and finally rated on the basis of their weighted mean scores, and presented in Table VI.

The majority of respondents (teachers and students) opined that, the knits treated with microcapsules through exhaust method at the 5th wash cycle possessed very strong (4.20/4.10) aroma intensity, followed by 0^{th} wash which exhibited strong (3.50/3.70) aroma intensity, 10^{th} wash knits possessed moderate (2.80) and strong (3.50) aroma intensity, 15^{th} wash with moderate (2.20/2.40) aroma and 20^{th} wash with mild (2.13/1.90) aroma intensity, respectively.

TABLE VI
OLFACTORY ANALYSIS OF VETIVER MICROCAPSULES FINISHED ORGANIC COTTON KNITS

Finishing methods	Wash cycles	Intensity of aroma (WMS)			
		Teachers	Rank	Students	Rank
Exhaust method	0	3.50	V	3.70	V
	5	4.20	II	4.10	II
	10	2.80	IX	3.50	VIII
	15	2.20	XIII	2.40	XI
	20	2.13	XIV	1.90	XV
Pad dry cure	0	3.40	VI	3.60	VI
	5	4.26	I	4.30	I
	10	3.13	VII	3.70	VII
	15	2.86	VIII	2.20	XII
	20	2.26	XII	2.00	XIV

Further, the group of respondents (teaches and students) rated that, 5^{th} wash knits treated with vetiver microcapsules by pad dry cure method possessed very strong (4.26/4.30) aroma intensity, followed by 0^{th} wash with strong (3.40/3.60) aroma, 10^{th} wash with moderate (3.17) and strong aroma (3.7), 15^{th} wash with moderate (2.86) and mild (2.2) aroma, and 20^{th} wash with mild (2.26/2.00) aroma intensity, respectively. In general, knits finished by pad dry cure method exhibited better aroma intensity even after the 20^{th} wash compared to knits finished by exhaust method. This may be due to the combined effect of the binder, i.e. acrylic binder helps to fix the finishing agents on the fabric with the application of pressure. Whereas, in the case of the exhaust method, citric acid was used as the cross linking agent which was found to be less effective than the acrylic binder. Irrespective of method of application, the concentration of fragrance on the fabric decreases with the increase of wash treatments due to the leaching of the finishing substrate from the surface of the fabric [15].

IV. CONCLUSION

The *Vetiver zizanoides* is a rich source of numerous phyto and bio active constituents namely, diethyl pathalate, Isopropyl dimethyl carboxylic acid, isoaromadendrene epoxide and beta-vetivenene etc., registered free radical scavenging, anti-inflammatory, UV protection, antimicrobial, antifungal and aromatic properties. The vetiver oil microcapsules possessed better morphological and thermal properties which are suitable for the finishing of textiles. The treated fabric possessed durable antimicrobial and aromatic properties even after several launderings. The vetiver oil microencapsulated organic cotton knits are free from hazardous chemicals and have bioactive compounds which not only serve as an infection control and barrier material, but also provides a soothing, cooling and pleasant odour and keeps the wearer healthier and comfortable. Hence, the eco-designed multifunctional knitted fabric is suitable for variegated health care textiles ranging from infant clothing to hospital clothing and hosiery.

ACKNOWLEDGMENT

The author acknowledges Directorate of Research, UAS, Dharwad for the encouragement and financial support.

REFERENCES

[1] S. Fijan and S. S Turk, Hospital textiles, are they a possible vehicle for healthcare-associated infections? *Int. J. Environ. Res. Public Health*, 2012, 9: 3330-3343.

[2] O. L. Shanmugasundaram, Antimicrobial finish in textiles. *The Indian Text. J.*, 2007, 52(3): 10-17.

[3] S. Mishra, K. Sharma, Mohapatra, S. and Chauhan, D., An overview on vetiveria zizanoides. *Res. J. Pharm. Biol. and Chem, Sci.,* 2013, 4 (3): 777-783.

[4] N. Azizi, N. Ladhari, Y.Chevalier and M. Majdoub, Study on perfume sustained-release from cotton fabric treated with polystyrene microspheres. *Int. J. Appl. Res. Text.*, 2014, 2 (2) : 42-51.

[5] A. Muresan, A. Cerempei, S. Dunca, R. Muresan and R. Butnaru, Aromatherapeutic characteristics of cotton fabrics treated with rosemary essential oil. *Cellulose Chem. Technol*., 2009, 4 (9-10) : 435-442.

[6] S. N. Rodrigues, I. Fernandes, I. M. Martins, V. G. Mata, F. Barreiro and A. E. Rodrigues, Microencapsulation of limonene for textile application. *Indian Engg. Chem. Res.,* 2008, 47 (05) : 4142-4147.

[7] L. V. Guardiola, Matrix-assisted concentration and microencapsulation of lime juice. *Ph. D. Thesis,* Uni. Illinois at Urbana-Champaign. 2011.

[8] S. F. Mulyaningsih, J. and Reichling, M. Wink, Antibacterial activity of essential oils from eucalyptus and of selected component against multidrug resistance and human pathogen. *Pharm. Biol.,* 2011, 49(9) : 893-899.
[9] K. C. Kaur, U. Bhardwaj, S. Kaushal and A. K. Sandhu, Chemical composition and biological properties of *Chrysopogon zizanioides* (L.) Roberty syn. *Vetiveria zizanioides* (L.) Nash- A Review. *Indian J. Natural Prod. Reso.,* 2015, 6(4) : 251-260.
[10] F. Mercela, C. Lucia, B. Eva, G. David, B. M. Angeles and B. Luis, Microencapsulation of essential oil by interfacial polimerization using polyurea as a wall material. *J. of Encaps. Adsorp. Sci*., 2015, 5 (4): 165-177.
[11] S. Mohanraj, P. Vanathi, N. Sowbarniga and D. Saravanan, Antimicrobial effectiveness of *Vitex negundo* leaf extracts. *Indian J. Fibre Text. Res.,* 2011, 37 (02) : 389-392.
[12] G. Thilagavathi and T. Kannaian, Combined antimicrobial and aroma finishing treatment for cotton, using microencapsulated geranium (*Pelargonium graveolens* L' Herit. ex Ait.) leaves extract. *Indian J. Natural Product Resource,* 2010, 1 (3) :348-352.
[13] M. P. Sathianarayanan, B. M. Chaudhari and N. V. Bhat, Development of durable antibacterial agent from ban-ajwain seed (*Thymus serpyllum*) for cotton fabric. *Indian J. Fibre Text. Res.,* 2011, 36 (3) : 234-241.
[14] K. Ramya and V. Maheshwari, 2015, Antiseptic treatment for human foot wounds using *Piper betel* extract finished bamboo/ cotton fabrics. *Indian J. Fibre Text. Res.,* 40 (02) : 213-216.
[15] A. Karolia and S. Mendapara, Imparting antimicrobial and fragrance finish on cotton using chitosan with silicon softener. *Indian J. Fibre Text. Res.,* 2007, 32 (2) : 99-104.