

EFFECT OF MICROENCAPSULATING OF DENIM FABRICS WITH HERBAL EXTRACT ON THE HANDLE AND CARE PROPERTIES

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Abstract : In recent years, handle and Care properties has become a major requirement in textiles. In the present study focuses on the micro-encapsulation to the 100 % cotton denim fabric using three herbal extracts of *Ricinus communis*, *Senna auriculata* and *Euphorbia hirta* to impart their antimicrobial efficiency by finishing the methanol extracts of these herbs on the denim fabric. The combinations of 1 part of *Ricinus communis*, 3 part of *Senna auriculata* and 2 part of *Euphorbia hirta* and conditions by ENISO20645 have been studied and the antimicrobial efficiency of these extracts (1:3:2) and the conditions of 20kgf/cm² pressure and 20m/min rpm speed are followed by pad dry cure method for best results. To enhance the durability of the finished fabric, treated and washed samples are tested using such test methods- Drape Coefficient (%) (IS 8357/1977) ,Air Permeability Test (IS 11056: 1984) etc ,SEM and FTIR .The result indicated that the drapability and crease recovery of the sample was found increased after washes along with air permeability increase.The maximum was noticed in washed samples. Further the stiffness was found predominantly reduced after washing, and no change noticed in water repellency. In the SEM analyzed it was clear that the microcapsules were present in interstices of the fiber assembly of fabric even after 30 washes proving the adherence of the finishing in the samples and in the FTIR the stretching frequencies were found to exist even after 10, 20 and 30 washes.

Key words: Antimicrobial activity, care properties, Denim fabric, Medicinal herbs, Micro-encapsulation and Wash durability .

1. Introduction

Clothes are considered as the second skin of the human kind. It plays a vital role after food, and before shelter. Textiles have such an

important bearing on our daily lives that everyone needs to know something about them. Since time immemorial, people have been using textiles of various types, from covering the body for modesty, for warmth, for personal adornment, to display of personal wealth. Eco-friendly products are highly beneficial to our health as also to the environment. The quality of our lives can be greatly improved with the use of these green products that are made from natural raw materials.

Denim is the most preferred clothing of today's youth. The spread of denim culture all over the world brought with it a trend of fast changing fashions and manufacturing technology. The healing value of herbal clothing and its usage is based on the principle of touch. By coming in contact with an herbal cloth, the body loses toxins and its metabolism is enhanced. Herbal clothes are completely free of synthetic chemicals and toxic irritants, and being totally organic, sustainable and biodegradable. Textiles are an excellent substrate for bacterial growth and microbial proliferation under appropriate moisture, nutrient and temperature conditions. Multi-functional finishing is defined as treating textile fabrics with two or more finishing agents in a combined bath, in a single step. Environmentally friendly materials were used for multi-functional finishes[1]. The increasing demand for multi functional fabric materials requires a strong multidisciplinary approach as well as the merging of traditional scientific disciplines[2]. A novel approach for imparting multi-functional properties, i.e., anti-crease, self-cleaning, UV-protecting as

well as anti-bacterial properties onto cotton fabric[3]. Micro encapsulation is a technique in which tiny droplets of benefit laden products such as moisturizers, fragrances, deodorizers ,vitamins or repellents are packed in microscopically small capsules sealed hermetically thereby preserving them. It is vital that microencapsules are stable and durable. The diameter of capsule varies from 1m depending upon the application; an area of 1sq cm would contain 1 million capsules[4]. Micro encapsulation has attracted the interest of the Dyeing, Printing and Finishing in textile wet processing method, for the last decade[5]. The objectives of the study are to screen for functional properties such as Antibacterial, Antifungal, Anti odor, Mosquito Repellency and Microencapsulation from selected herbs, To optimize the effective herbal combination of the functional property of the selected finishes, To finish the optimized herbal combination on the 100% cotton denim fabrics and to evaluate the handle and care properties, SEM and FTIR before and after wash of the finished fabrics.

2. Materials and methods

2.1 Materials

The fabric was sourced from the market with respect to the expected quality requirements. The fabric chosen for the study are 100% cotton denim fabric, square weight-219.4(Twill weave) The above samples were pre treated with hot water before finishing.

2.2 Selection of herbs

For this research, twenty healthy and live herbal plant parts were selected. The botanical names of the selected herbs are as follows: Ricinus Communis (leaves), Ricinus Communis (seeds), Abutilon indicum (leaves), Solanum surattense (Leaves), Coccinia grandis (Fruits), Coccinia grandis(leaves), Datura metel (leaves with fruits), Aleo vera (flower) , Aleo vera (leaves), Cardio spermum halicacabum (Leaves), Cissus quadrangularis (whole plant), Albizia amara (Leaves), Leucas aspara (stem, leaf and flower), Euphorbia hirta (mixture of stem, leaf and flower), Kathari flower,Cereus janacars (whole plant),Tribulus terrestris (Whole plant), Senna

auriculata(Leaves),Passiflora foetida-(Stem, Leaf and flower)and Poolapoo(whole plant) were collected from different regions in and around Coimbatore district, which were authenticated by the Botanical Survey of India. These herbs were selected because they exhibited antimicrobial activity and tannic properties.

2.3 Extraction process of herbs

The extraction process was done in three stages, such as drying, grinding and extraction. Two types of extraction methods were followed: viz methanol and aqueous extraction was done for all the twenty selected herbs.

2.4 Finishing method

The selected denim fabric samples were finished with the herbal plant extraction by dip method and analysis of antibacterial activity by ENISO 20645 method was followed to find out the three best activity herbs.

2.5 Optimized parameters

After conduct of Pilot study, the following parameters were selected. Three herbs such as **methanolic extract of Ricinus communis**, **methanolic extract of Senna auriculata** and **aqueous extract of Euphorbia hirta**, in the proportion of (1:3:2) was taken and conditioned with **20Kg/cm² Pressure**, **20m/min rpm**, followed by pad dry cure method.

2.6 Micro encapsulation of fabric by ionic gelatin process

2.6.1 Preparation of Microencapsules

The microencapsules were applied on the fabric by pad dry cure method.

Recipe

Denim Fabric meters	-	4
Herbal powder (core) grams	-	240
Material liquor ratio	-	4:4
Sodium alginate (wall)	-	3%
Time hours	-	2
Temperature	-	45 °C

Solvent -
Calcium chloride

Procedure for Micro encapsulation

Microcapsules containing extracts of herbal combinations 1 Part of **Ricines Communis**, 3 Parts of **Euphorbia Hirta** and 2 Parts of **Senna Auriculata** as core material and sodium alginate as the wall material was prepared.

Ten grams of wall material was allowed to swell for half an hour by mixing with 100 ml of hot water. To this mixture 50 ml of hot water was added and stirred for 15 minutes maintaining the temperature between 40°C and 50 °C. to this mixture, 10 ml of core material sodium alginate was added and this was transferred to a centrifuge and rated at 300-500 rpm speed for 15 minutes. This was sprayed into 2% of calcium chloride solution by means of a sprayer. The droplets were retained in calcium chloride collection bath for 15 minutes. In this bath the calcium ions diffused with the alginate solution, thereby hardening the matrix and forming a solid hydro gel system. The microcapsules were obtained by decantation and repeated washing with isopropyl alcohol followed by drying at 45 °C for 12 hours. The microcapsules were then used for finishing on the selected fabrics with pad dry cure method.

2.7 Finishing of selected fabrics with pad dry cure method.

Recipe

Denim fabric	-	4
Meter (100% Cotton)		
Microcapsules	-	2%
Binder (Citric acid)	-	8%
Temperature	-	55°C
Time	-	30
minutes		

The basic process was that the microencapsules were compounded at a certain ratio and padded on the fabric .The microencapsules were attached to the fabric with binder. By evaporating water during the drying process in a tenter, it allows only the

required ingredients to combine with the fabric.

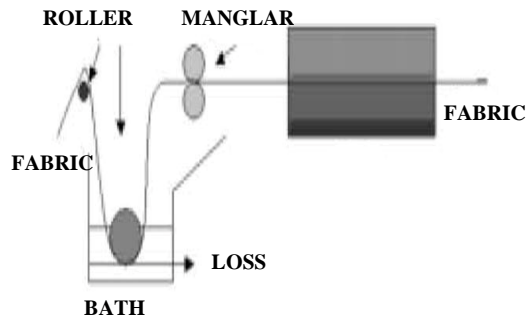


Figure 1: pad dry cure process

The Compounded solution consisted of 2% of the microencapsules with binder citric acid 8% and normal textile chemicals were added, such as softener, anti-static electricity agent and was kept in a bath. The fabric was dipped into the compounded solution using a roller. The fabric was sent through a padding mangle to squeeze the dipped chemicals out of the bath at the ratio of the pick up rate when the fabric was passed between the rollers. The heat dryer dried the fabric with heat for evaporating water and maintaining its width. The encapsulation worked inside the fabric, filling the spaces between the fibers with an ultra thin film of polymer creating a permanent barrier that was breathable, yet impermeable to both water and wind.

3. Evaluation of the handle and care properties

3.1 Drape Coefficient (%) (IS-8357/1977)

The **Drape Coefficient [(%) (IS-8357/1977) (Plate -28)]** was used to measure the drape of the fabric. The drape tester was placed firmly on a level table. The light was switched on. The specimen holder was removed from the bayonet socket and a cut fabric specimen was placed between the plates. The stub of the specimen holder assembly was held, and briskly moved up and down ten times, and tested on the table each time, for a moment. A square of ammonia process paper on the base of the instrument was placed flat. The stub of the specimen holder was inserted in the socket

on the threaded bolt, pressed upwards and turned counter clockwise to lock the holder in position. Looking along the level of the base board, the height of the drooping edge of the drape specimen was adjusted, so that the lowermost edge was just above the paper without touching it. The setting knob of the timer was adjusted for the required time of exposure. At the end of the exposure time, the ammonia paper was removed, and placed in a developing box containing a few millimeters of strong ammonia solution. When the latent was developed, the paper was removed. The paper was conditioned to moisture equilibrium in standard atmosphere. The drape pattern was cut out with a pair of scissors and its mass determined in gram weight, exact to two decimal places.

Mass per unit area of the paper was determined, by cutting a known area of the original paper and weighing. The specimen was reversed and the drape pattern obtained with the other surface upwards.

The following formula was used to calculate the percentage of drape coefficient.

$$\text{Drape co-efficient (F \%)} = \frac{W/w-a}{A-a} * 100$$

Where, mass of drape pattern (gms) = w

Mass/unit area of the paper = W

Area of circle of 12.5 cm diameter = a

Area of circle of 25 cm diameter = A

Drape was the ability of a fabric to fall under its own weight into wavy folds of different nature. Fabric drape can be evaluated objectively as well as subjectively. The same procedure was repeated for five samples and the mean value was calculated.

3.2 Air Permeability Test (IS 11056: 1984)

A portion of the conditioned specimen was mounted between the clamp and the circular orifice with sufficient tension to eliminate wrinkles, if any and care taken to ensure that the fabric was not distorted. The suction fan or other means to force air through the fabric was started; the rate of the flow of air was adjusted till a pressure drop of one centimeter water across the fabric was indicated. The rate of air flow was noted in cm^3/s . The test was repeated at different places. The same

procedure was repeated for five samples and the mean value was calculated.

3.3 Crease Recovery angle (IS 4681: 1981)

The testing equipment was leveled with the help of leveling screws and the spirit level. Level the testing equipment with the help of leveling screws and spirit level. The specimen was folded end to end in half, with its edges gripped in a line with the help of tweezers, no more than 5 mm from the ends the folded specimen was placed on the plate of the loading device and load was applied gently without delay. The load was removed after 5 minutes. The removal of the load should be done in 0.5 seconds. Half of the specimens were folded face to face and the other half back to back. In order to mount, the one limb of the specimen was placed in the clamp of the instrument and the other held by the tweezers, in such a manner to ensure that there was hardly any disturbance. The specimen was thus held for at least one minute before the angle was measured. While the specimen was held by the clamps, adjustments were made such that the suspended limb of the specimen is always in a vertical position or horizontal position depending upon the type of instrument used. The reading of the crease recovery angle was taken after 5 minutes after the load was removed.

The angle of recovery was measured for all the warp way and weft way specimens folded face to face and back to back in the same way. The same procedure was repeated for five samples and the mean value was calculated.

3.4 Stiffness to fabric (IS 6490:1971)

Method for determination of stiffness of fabrics as denoted by Indian standard, 1971. The tester was placed on a table or bench so that horizontal platform and inclined reference line were at eye level of the operator. The platform was adjusted, with the help of a spirit level so that it was horizontal.

One of the specimens was placed on the platform with the scale on top of it lengthwise and the zero of scale coinciding with the leading edge of the specimen. The scale was held in the horizontal plane, the specimen was

pushed, and the scale slowly and steadily moved, when the leading edge projected beyond the edge of the platform. An increasing part of the specimen would overhang and start bending under its own weight. If the specimen had a tendency to twist, a reference point was taken at the center of the leading edge. The specimen which twisted more than 45 degree was not measured. The length of the overhanging portion was noted from the scale to the nearest millimeter. five readings from each specimen was taken, with each side up, first at one end and then at the other. Similarly, at least five test specimens for each warp way and weft way was tested. The weight per unit area of the fabric was determined, according to IS: 1964-1971 and expressed in terms of milligrams per square centimeter. Alternatively the weight per unit area could be determined by weight of all the warp way and weft way test specimens together, after completion of stiffness test. The same procedure was repeated for five samples and the mean value was calculated.

3.5 Water Repellency spray test (AATCC22)

Water Repellency spray test (AATCC22) was used to test the fabrics. Before testing was carried out, the specimen should be conditioned for at least 24 hours in the standard atmosphere. Water sprayed against the taut surface of a test specimen under controlled conditions, produced a wetted pattern whose size depended on the relative repellency of the fabric. To carry out the test, test specimens were fastened securely in the metal hoop of the water repellency tester so that it represented a smooth wrinkle free surface and placed face up on the tester.

The metal hoop was adjusted, so that the centre of the spray coincided with the centre of the metal hoop. Later, 250 ml of distilled water at normal temperature, was poured into the funnel and the whole quantity sprayed on

the test specimen for a period of 25 - 30 sec. (AATCC standard).

Then, the metal hoop was detached from the stand, and confirmed that water had penetrated to the back of the test specimen. With the face side of the test specimen down, the metal hoop was held by one edge and the opposite edge tapped lightly once against the table. Then it was rotated 180°C and similarly tapped again once on the point previously held, to remove any excess water drop. The final step was to compare the wetting of the test specimen with a photographic rating standard and grade accordingly. The same procedure was repeated for five samples and the mean value was calculated.

3.6 Wash Durability Test

The microcapsules finished denim fabric were analyzed for their wash durability by subjecting the sample to washing and testing their antibacterial efficiency. The denim fabric was subjected to washing by industrial machines and the antibacterial activity of the washed fabric was assessed by ENISO 20645 test method.

3.7 Scanning Electron Microscopy Test

The scanning electron microscopy was used for confirming the binding of microcapsules and alignment on to the fabric sample. The SEM photographs of microencapsulated, and washed samples were examined under X 7000 magnifications

3.8 Fourier transforms infrared spectroscopy Test

To ascertain the activity of herbs in micro encapsulation and after 10, 20 & 30 washes of the fabrics, FTIR spectrum were recorded on shim at 2u.FTIR-8000/400 S modal spectrometer in the regain 4000-400 cm⁻¹ using kbr discussing FTIR spectra the samples were analyzed for their variation in chemical groups.

3.9 Nomenclature

Table 1

S.No	Fabric samples	Fabric samples	Abbreviations
1	MD	Microencapsulated sample (finished sample)	100% cotton
2	MD10	Microencapsulated sample after 10 washes (washed sample)	100% cotton
3	MD20	Microencapsulated sample after 20 washes (washed sample)	100% cotton
4	MD30	Microencapsulated sample after 30 washes (washed sample)	100% cotton

4. Result and Discussion

4.1 Drape co-efficient (%)

The following figure -2 depict the Drape co-efficient (%) of microencapsulated finished fabrics.

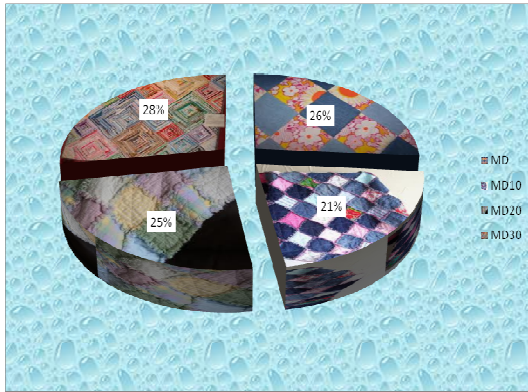


Figure 2 : Drape co-efficient (%) of Microencapsulated finished fabrics

Figure 2 represent the drape coefficient of the microencapsulated samples which was maximum in MD30 at 5.73 %, followed by samples MD20 (6.11 %) and MD10 (17.7 %.) Hence it could be concluded that drapability increased in finished and washed samples.

4.2 Air permeability ($m^3/cm^2/sec$)

The following table 2 shows the Air permeability ($m^3/cm^2/sec$) of Microencapsulated finished fabrics

Table 2 Air permeability ($m^3/cm^2/sec$) of Microencapsulated finished fabrics

S. No	Fabric samples	Air permeability ($m^3/cm^2/sec$)	
		Mean	% loss or gain
1	MD	18.1	-
2	MD10	14.3	-20.9
3	MD20	21.9	20.9
4	MD30	30.4	67.9

Table 2 represent the air permeability ($m^3/cm^2/sec$) of sample MD which was 18.1, it decreased after ten washes to 20.9 % and increased to 67.9 % in sample MD30, whereas the increase in sample MD20 was 20.9 % . Hence it could be concluded that after several washes, the air permeability of the fabric increased because of the removal of starch, finishes from the pores of the fabric.

4.3 Crease recovery angle (degrees)

The Following Figure 3 depict the Crease recovery angle (degrees) of Microencapsulated finished fabrics

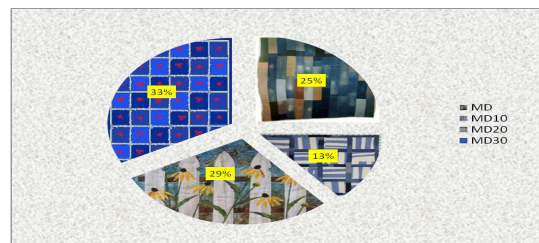


Figure 3: Crease recovery (degrees) of Microencapsulated finished fabrics

Figure 3 represent the Crease recovery angle (degrees) of microencapsulated samples

exhibited by MD10 at 47 %, but increased in samples MD20 (14.5 %) and MD30 (29.1%) over unwashed sample. Hence it could be concluded that the samples subjected to thirty washes had the maximum Crease recovery over the original sample.

4.4 Stiffness (mg-cm)

The Following figure 4 depict the Stiffness (mg-cm) of Microencapsulated finished fabrics.



Figure 4: Stiffness (mg-cm) of microencapsulated finished fabrics

Figure 4 show the stiffness of the finished sample MD, which was observed to be 2787 (mg-cm) .This, was increased by 6.99 % in sample MD10, but decreased in samples MD20 and MD30 by 5.84 and 45.4 % respectively.

Hence it could be concluded that the stiffness reduced predominantly in the fabric subjected to several washes.

4.5 Water repellency -Spray rating

The following Table 3 expresses the Water repellency Spray rating of Microencapsulated finished fabrics

Table 3 Water repellency -Spray rating of Microencapsulated finished fabrics

S.No	Fabric samples	Water repellency Spray rating
		Mean
1	MD	50
2	MD10	50
3	MD20	50
4	MD30	50

Table 2 clearly depicts that all the samples of MD,MD10,MD20 and MD30 exhibited water repellency spray rating to 50 ISO-1 expressing

the same value as the finished and washed samples for Water repellency property test.

4.6 SEM analysis for the Microencapsulated sample

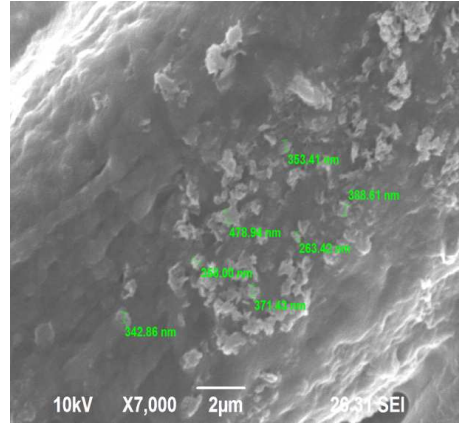


Figure MD

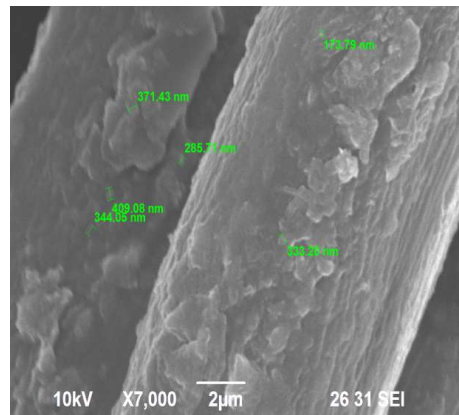


Figure MD10

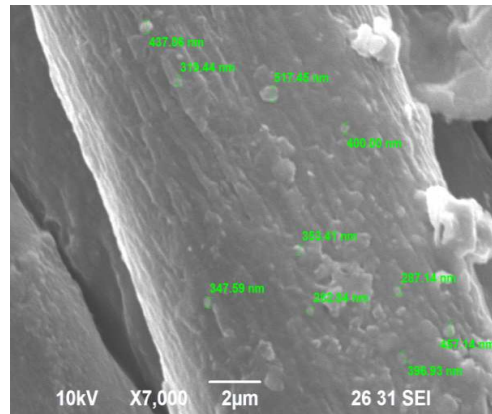


Figure MD20

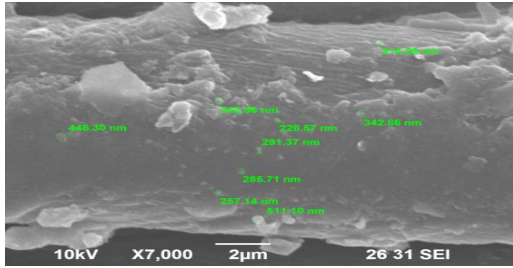
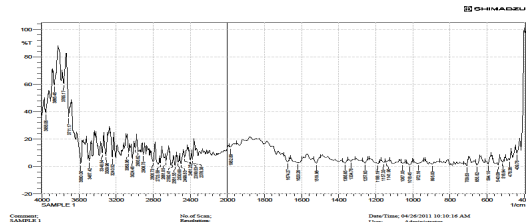


Figure MD30

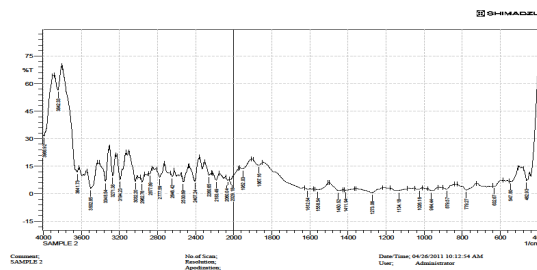
Figure 5: SEM analysis for the Microencapsulated sample

The SEM photographs are shown in figure 5 at x7000 magnification level. It is clear that the microcapsules are present in interstices of the fiber assembly of fabric even after 30 washes proving the adherence of the finishing in the samples.

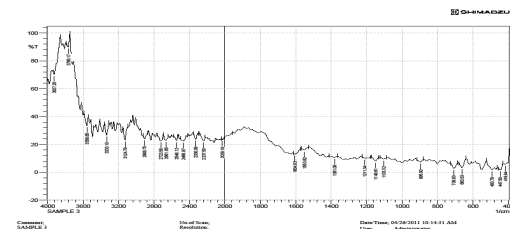
4.7 FTIR analysis for the Microencapsulated sample



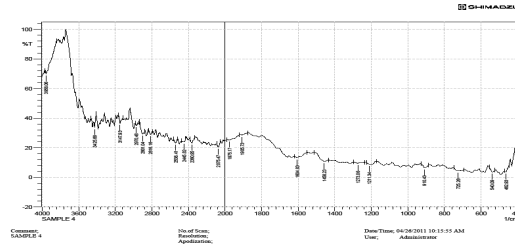
Microencapsulated finished fabric (MD)



Microencapsulation finished fabric after 10 washes (MD10)



Microencapsulation finished fabric after 20 washes (MD20)



Microencapsulation finished fabric after 30 washes (MD30)

Figure 6: FTIR analysis for the Microencapsulated sample

The Microencapsulation technique strongly adhered to the fabric, which shifted the frequency significantly. Moreover, most of the stretching frequencies were found to exist even after 10, 20 and 30 washes. This inferred that there was a binding between the cellulose fiber and the added microencapsules in fabrics .Hence it is found to be present even after many washes. This is strong evidence that herbal extracts were imparted in the fabric.

5. Conclusion

Plant kingdoms consist of millions of species. The uses of these plants to the human beings are innumerable .It is highly impossible for the human kind to enumerate the usefulness of the plants and its various parts. Many plants contain plenty of medicinal properties present in them. Among these, twenty commonly available plant species were selected for the study. There were natural inhibitors present in it. These characters were identified in the selected plant species and the extracts were collected. After a preliminary study, 3 plant species with desirable characters were short listed. The herbal extracts from these plants were applied on the denim fabrics by three different application methods i.e. finish. The finishes selected were microencapsulation. The finished fabrics were subjected to handle and care properties to identify the best finish. The adherence and long lasting nature of the finish was also tested with the SEM analysis and FTIR analysis. After the procedure it was identified by the investigator that the finishing technique by the microencapsulation method in the fabric

sample 100% cotton denim fabric (sample D) possessed all the expected characters like durability of the fabric, increased fabric thickness, weight, drapability, luster, evenness and antimicrobial activity. In turn the above characters enhanced the wearing capacity of the denim fabric for an extended period without any skin/dermal irritations/infections, without any bad odor and protection from mosquitoes.

6. References

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