Indian Journal of Fibre & Textile Research
Vol 43, September 2018, pp. 375-380

Short Communications

Performance of chitosan: jasmine oil microcapsule on jute fabric

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Received 16 March 2017; revised received and accepted 23 August 2017

Chitosan: jasmine oil based microcapsule has been prepared by coacervation method and then applied on bleached jute fabric by pad-dry-cure method. The performance of fragrance finished jute fabrics has been assessed by physical properties, subjective assessment, scanning electron microscope, percentage moisture regain (%), fourier transform infrared spectrum, durability against home laundering and handle property. Morphological studies confirm that the chitosan: jasmine oil microcapsule of 100 micron dimension has formed. Performance properties show that the chitosan: jasmine oil microcapsule can be fixed on the surface of jute fabric and the fragrance is sustained up to five home laundering.

Keywords: Chitosan, Jasmine oil, Jute, Microcapsule

Functional finishing is used to enhance the performance of a textile product and each finish differs from others, depending on their end uses. Jute fibre is one of the important natural fibres, and mainly used for hessian, sacking, yarn, carpet backing cloth, handicrafts, ropes and cords. Presently jute based home textiles are preferred in an international market due to its eco-friendliness. The consumption of jute products can be further enhanced by imparting functionality like flame retardant, fragrance and softening finishing[1].

Microencapsulation technology is one of the important finishing processes and is used to prepare tiny containers of an active core compound and a solid shell. Solid shell of the microcapsule releases the active core contents under controlled conditions to impart functional properties like fragrance and antimicrobial effects on textile products. Generally, the active core substance is an organic compound and can be released by controlled UV radiation (for curtain textiles), change of temperature (for sweat odour reduction), mechanical impact (by friction and/or pressure), dissolution of polymer wall and diffusion through polymer wall. There are so many methods available to produce microcapsule in which the basic concept is the coating of a thin polymer on small solid particles or liquid droplets [2,3].

Fragrance finishes on cotton textiles are mainly achieved by using microencapsulation technology, since it shows durable functionality [4,5]. For fragrance finish, essential oils generally have been used due to their eco-friendly nature. Melamine–formaldehyde, gelatin–arabic gum and chitosan microcapsules containing essential oils have been applied to cotton textiles[6-9]. Jasmine oil: melamine-formaldehyde microcapsule was prepared by *in situ* polymerisation method and applied on jute-cotton blended fabric in presence of a cross-linker. Results inferred that partial de-lignified jute fabric has improved the durability of the fragrance finishing[10]. Vanillin:chitosan microcapsule was prepared by spray-drying method with 51% encapsulation efficiency and applied on cotton fabric with citric acid as a cross-linking reagent. This microencapsulated cotton fabric retained nearly 22% of fragrance under 65°C and 80% humidity condition [5].

Chitosan, citric acid and silicon softener combination was applied on cotton fabric to impart antimicrobial, wrinkle recovery and fragranceproperties[11]. Neem and Mexican daisy microcapsules were prepared by simple coacervation technique with gum acacia as wall material and applied on cotton fabric. This microencapsulated cotton fabric shows good antimicrobial activity against *Staphylococcus aureus* and *Escherichia coli* up to 15 home laundering[12]. β-cyclodextrin is a biopolymer and forms an inclusion complex with different organic compounds which have different industrial applications[13]. β-cyclodextrin is modified with monochlorotriazine (β-CD-MCT) and fixed on cotton fabric for entrapping sandalwood oil. Results found that sandalwood fragrance was retained in β-CD-MCT-treated cotton fabrics for 21 days, while control cotton fabric retained for 8 days[14].

Patchouli oil based microcapsule was prepared by complex coacervation procedure and fixed onto

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cotton fabric through 1, 2, 3, 4-butanetetracarboxylic acid (BTCA). Results indicated that the 50% patchouli oil was retained in the microcapsules due to chemical cross-linking between BTCA and cotton fibre, which improved the performance of the fragrance finishing [15]. Methanolic extract of medicinal herbs was microencapsulated with gum acacia by coacervation technique and applied on cotton fabric by pad-dry-cure method using a cross-linking agent. It was inferred that copper-enriched and herbal based microcapsule showed good antimicrobial activity on cotton textiles and retained the activity up to 10 home laundering[16,17].

Literature review revealed that the microcapsule is mainly prepared from synthetic polymers through emulsion polymerization method; followed by its application using a cross-linking agent or a polymer for its coating on the surface of the fibre. The slow and sustained release of the fragrance mainly depends on the cross-linking efficiency and the nature of shell polymer. An attempt has been made to prepare the chitosan:jasmine oil microcapsule by the conventional method and applied on a bleached jute fabric; the performance properties were evaluated in terms of SEM, FTIR, *K/S* value, handle properties and whiteness index.

Experimental

Material

Grey jute fabric with 60 ends/dm, 52 picks/dm and 250 g/m^2 GSM was used for this study. Jasmine oil was procured from the local market of Kolkata and used without any purification. Chemicals used in this study were of LR Grade.

Bleaching of Jute Fabric

Jute fabric was bleached with hydrogen peroxide as per conventional method in order to improve its whiteness. After bleaching, the fabric was washed with hot water, cold water, neutralised with dilute acetic acid solution, rinsed with water and dried at ambient condition.

Preparation of Chitosan: Jasmine Oil Microcapsule

Chitosan: Jasmine oil microcapsule was prepared as per the following procedure[9].Two millilitre of jasmine oil was added in 20 mL of 0.5 wt% chitosan solution along with 0.5 wt% of non-ionic surfactant and the solution was stirred at 700 rpm for 10 min. The emulsion was dripped into solutions containing 1% of sodium hydroxide, stirred at 100 rpm for 10 min and then kept for another 30 min. Then, the solution was filtered, washed three times with distilled water and dried at 30°C for a period of 15 h in order to remove residual moisture.

Release Behaviour

A known amount of microcapsules and fragrance oil was placed separately in an oven at 35°C for the period of 8 h in order to assess the release of jasmine oil from microcapsule. The final weight of microcapsule and fragrance oil was collected and the data was then expressed in terms of percentage weight loss[9].

Fragrance Finishing

Micro capsule stock solution (10 gpL) was prepared and the *p*H of the stock was adjusted to 6.0 by adding dilute acetic acid solution. Bleached jute fabric of dimension 30 cm × 30 cm was impregnated in the stock solution for 5 min and padded with 95% expression using a laboratory padding mangle. After padding, the fabric was dried at 70^{o}C for 10 min.

Characterisation

Subjective Assessment of Fragrance

Fragrance of the finished jute fabrics was evaluated by a subjective assessment method under a standard atmospheric condition (25±2^{o}C and 65±5% RH) without any wind. Each finished sample was packed in an airtight polythene bag in order to prevent the release of fragrance. A panel of 16 judges was asked to rate each fragrance finished fabric on a scale of 1 (no fragrance) to 10 (excellent fragrance). Judges were allowed to receive 3-4 smells for each sample in an open corridor and asked to rank the fragrance from 1 to 10 scales. Intermittently, coffee beans were used to neutralize the smell of previous sample. The overall fragrance grade of each sample was calculated as per the following equation [11, 18]:

$$\text{Fragrance grade} = \sum_{i=10}^{10} \frac{S_i\, n_j}{10n} \times 100$$

where n is the total number of judges; S_i, the judge's rating ; 10, the highest rating; and n_j, the number of judges giving rating S_i.

For the assessment of durability of the fragrance, finished sample was kept in an open atmospheric condition (20±10^{o}C; 65±5% RH) for the period of 21 days. The fragrance of the samples was subjectively assessed on alternate days and the mean value of three samples was taken.

Handle Property

Shirley stiffness tester was used to evaluate the handle properties of samples in terms of bending length, bending length flexural rigidity and bending modulus as per standard [19].

Washing Durability

The washing durability was performed as per AATCC 61(2A)-1996 test method and three fragrance finished jute fabrics were evaluated for each experiment. One washing process of this method is equivalent to five cycles of home laundering [20].

CIE Whiteness Index

Premier Colorscan spectrophotometer (Model SS6200) at illuminant $D_{65}/10^o$ observer was used to determine whiteness index (HUNTER Scale), yellowness index (ASTM D 1925) and brightness index (TAPPI 452). Each fabric was folded twice and measured four times at different portions of the fabric surface and the average value was recorded.

SEM Study

For surface morphological study, microcapsule samples were magnified in JEOL scanning electron microscope of Model JSM 6360.

FTIR Study

Bruker FTIR analyzer of Model Alpha using an attenuated total reflectance (ATR) mode was used to derive FTIR spectra.

Results and Discussion

Optical Property

Generally, colour of raw jute fabric varies from golden yellow to dust grey colour and it depends mainly on retting process, residual minerals present in the fibre, added impurities during spinning and quality of the jute reed. Table 1 shows that this jute fabric has less than 50 whiteness index with high yellowness index (53); while after peroxide bleaching, the whiteness index of the fabric is significantly improved from 49 to 81. It is due to removal of added and natural impurities by the alkaline scouring and subsequent decolouration of natural colouring matters by alkaline peroxide. Intermittent acidic treatment can also demineralised the minerals present in the jute fibre.

Table 1 — Optical properties of raw and processed jute fabric

Jute fabric	Whiteness index	Yellowness index	Brightness index	K/S value
Raw	48.7	53.0	18.6	2.48
Scoured	44.1	52.0	15.5	3.08
Scoured & bleached	80.9	23.7	57.9	0.32

Jute is a multi-cellular fibre and has stiffness due to low micro fibril angle (<10^o). Ultimate cell in the jute fibre is mainly responsible for the stiffness of the grey jute fabric (>5.0cm in both directions). Table 2 shows that during scouring and subsequent peroxide bleaching, the cementing agent present in the jute fibre (hemicellulose) is partially removed and it relaxes the ultimate cell of the jute fibre, thereby reducing the stiffness property of the jute fabric (<4.0cm in both directions).

Scouring and bleaching processes also lead to weight loss of the jute fabric due to removal of natural and added impurities. It also decreases the GSM of the scoured/ bleached jute fabric and is mainly responsible for the drastic reduction in the bending modulus and flexural rigidity. The reduction in bending modulus due to scouring and bleaching on jute fabric as well as respective flexural rigidity are 63 & 81% and 63 & 74% respectively.

Jute is a natural composite fibre and hemicellulose portion binds the cellulose and lignin polymer. It is reported that hemicellulose is partially removed during scouring and bleaching that leads to defibrillation in the jute fibre. Bleached jute fibre is found finer than the grey state and so the dissipation of tensile load in each ultimate cell is more in bleached fibre than in untreated jute fibre. This factor is responsible for the reduction in tenacity of the jute fabric after scouring and bleaching by 6% and 16% respectively.

Property of Microcapsule

Coacervation method is used to prepare the chitosan: jasmine oil based microcapsule in which chitosan could precipitate in an alkaline solution.

Table 2 — Physical properties of raw and processed jute fabric

Fabric	Bending length, cm		Bending modulus, kg/cm^2		Flexural rigidity, mg.cm		Tenacity, cN/tex	
	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft
Grey	5.1	5.5	66.3	82.8	3046	3804	4.9	5.4
Scoured	3.4	4.0	22.0	33.3	1011	1530	4.5	5.2
Scoured & bleached	3.2	3.2	13.7	13.8	890	894	4.2	4.4

Sodium hydroxide solution forms coacervate drops by blowing a chitosan solution. In coacervation method, by varying the ratio of the chitosan and sodium hydroxide solutions, the diameter, porosity and strength of the microcapsules can be controlled[21]. After preparation, it is observed that these microcapsules are in non-spherical shape with a rough surface. Microscopic images show the presence of small pores in the chitosan walls which allow the controlled release of the encapsulated jasmine oil.

Release Behaviour

This experiment was conducted to measure the releasing behaviour of the jasmine oil with and without microcapsulation in terms of percentage weight loss. As expected, jasmine oil shows higher weight loss due to its volatility (82%) than microcapsules (15%). The results infer that chitosan biopolymer has potential to release the jasmine gradually[9]. FTIR spectra of different samples are taken in order to assess the release kinetics of the jasmine oil (Fig.1).

Jasmine oil is a main derivative of methyl dihydrojasmonate with alcohols, esters, ketones and aromatic compounds (eugenol and eugenyl acetate). The FTIR spectrum of the jasmine oil confirms the presence of O-H stretching, H-bonding due to alcoholic and phenolic groups at 3350 cm^{-1} , >C-H stretching from alkane groups at 2974 & 2927cm^{-1} respectively, >C=O stretching bond from aldehyde groups ; >C=C< stretching from alkenes at 1679cm^{-1}and >C=C< stretching from aromatic ring between 900 cm^{-1} and 675 cm^{-1}. Similarly FTIR spectra of chitosan show peaks responsible for alkane group (2915 cm^{-1}), amide groups (1650 and 1550 cm^{-1}), -C-N groups (1373cm^{-1}) and glucopyranose ring (1065 and 1028cm^{-1}) [22-23].

After formation of chitosan: jasmine oil based microcapsule; the peaks responsible for jasmine oil are drastically reduced in the microcapsule in comparison with only jasmine oil. It confirms the capsulation of the jasmine oil in the shell of chitosan polymer. After being exposed to releasing condition, the microcapsule retains the main peaks responsible for jasmine oil.

Assessment of Fragrance

The fragrance grade by subjective assessment, percentage moisture regain and whiteness index of bleached and CHT: JO microcapsule finished jute fabrics (before and after home launderings) are given in Table 3. Bleached jute fabric does not have any odour / fragrance due to removal of the added and adhered impurities from the jute fabric during alkaline peroxide bleaching process.

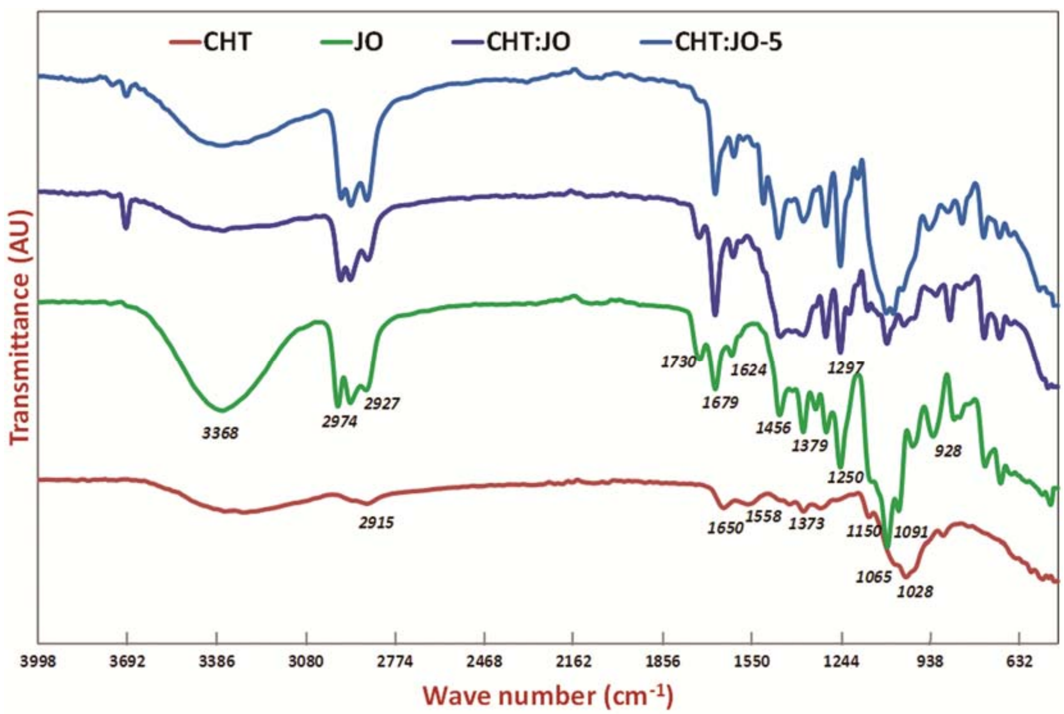

Fig. 1 — FTIR spectra of chitosan (CHT) , jasmine oil (JO), chitosan: jasmine oil microcapsule (CHT:JO) and chitosan: jasmine oil microcapsule after 5 days exposure (CHT:JO-5)

Table 3 — Fragrance grade, percentage moisture regain(%) and whiteness index of bleached and fragrance finished jute fabrics

Sample	Fragrance grade (Mean±SE)	% Moisture regain (Mean±SE)	Whiteness index (Mean±SE)
Bleached jute fabric	0.8±0.1	10.5± 1.0	80.4±4.2
Fragrance finished fabric after 0 wash	8.9±0.1	10.8±0.9	80.3±3.4
Fragrance finished fabric after 5washes	1.4±0.2	10.4±1.3	75.4±4.1

After fragrance finishing, obviously all judges give an excellent grade to the finished jute fabric (0 washes), while after 5 washes the fragrance grade is significantly reduced from 8.9 to 1.4. All judges felt that residual fragrance is perceived after 5 home laundering. The surface morphology of the chitosan: jasmine oil microcapsule finished jute fibre before and after home laundering is studied by scanning electron microscope.

Figure 2 confirms the presence of CHT: JO microcapsules uniformly on the surface of the jute fibre. However after five laundering, it is evidenced that microcapsules have been discharged from the surface of the jute fibre. It is well known that chitosan polymer has only weak hydrogen bonding with cellulose / lignin polymer so that chitosan: jasmine oil microcapsules could easily discharge from the surface of the jute fibre during home laundering.

The durability of the fragrance from the finished jute fabric against the environmental condition is also studied and the fragrance grade with respect to number of days is evaluated. It is inferred that core fragrance of the microcapsule gradually liberated to the surrounding atmosphere is due to the collapse of the shell polymer by change in atmospheric pressure and temperature [24].

Figure 3 indicates that the fragrance grade of the finished jute fabric is significantly decreased with increasing the number of days, when exposed under the standard atmospheric conditions. It is also revealed that this fragrance finish could be sustained in the open atmospheric condition for only one week. Judges felt that bleached jute fabric enhances the aesthetic appeal of the interior textiles, and fragrance finish further enhances the psychological preference of the material for the selection of the home textiles.

Chitosan: jasmine oil based microcapsule of 100 micron dimension has been prepared by the

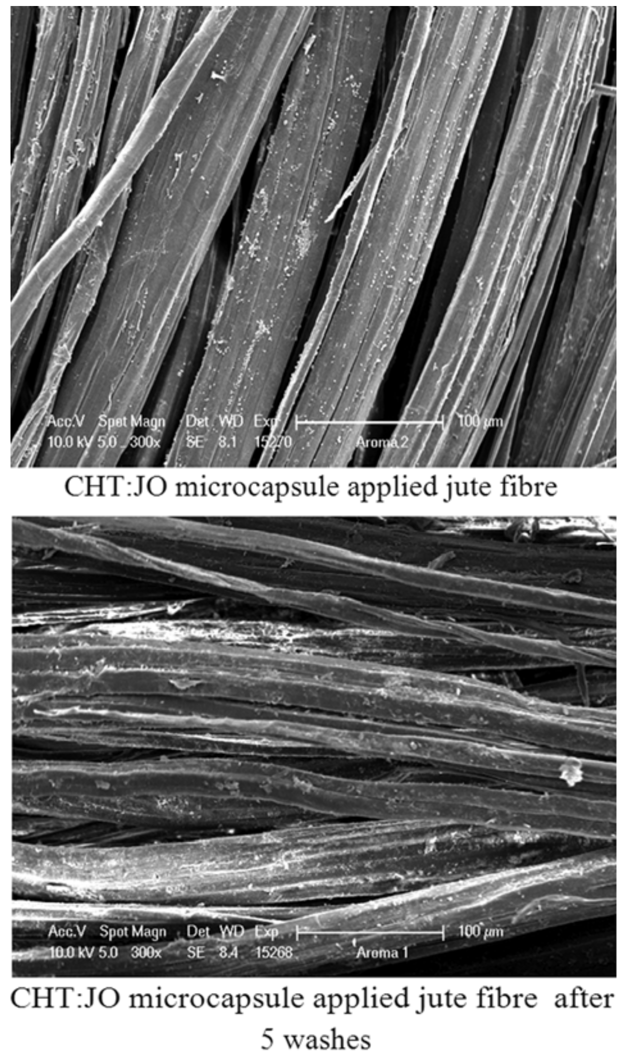

Fig. 2 — SEM photographs of jute fibres

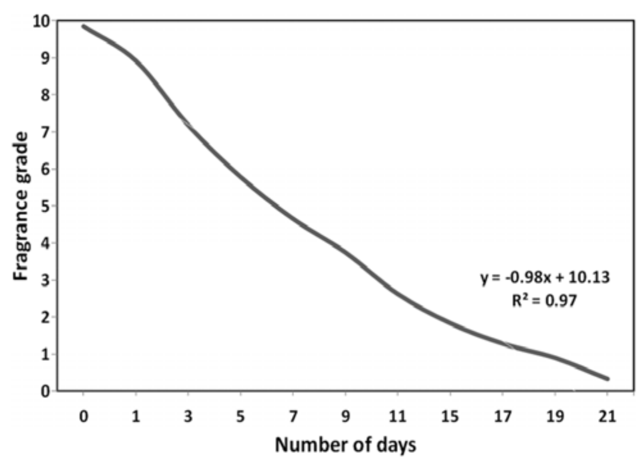

Fig. 3 — Fragrance grade of fabric with respect to number of days

coacervation method and it is applied on bleached jute fabric by pad-dry-cure method. Results reveal that CHT: JO microcapsule has potential to impart fragrance finish on jute textiles, however the washing durability is moderate due to less interaction between chitosan polymer and jute fibre. Further studies are in progress in order to improve its durability in jute fibre.

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