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Flame Retardant and Antimicrobial Ligno-cellulosic Fabric using Sodium Metasilicate Nonahydrate

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Flame retardant and antimicrobial functionalities were imparted in ligno-cellulosic jute textiles using sodium Metasilicate nonahydrate (SMSN), commonly known as "water glass". SMSN was applied on a jute fabric in different concentrations by padding method followed by drying. Flame retardancy of the fabric was evaluated by Limiting Oxygen Index (LOI) and by the burning behaviour, including the char length under a vertical flammability tester. The burning rate was found to decrease by almost 10 times after application of 2% SMSN compared to that observed with the control jute fabric. Thermogravimetry (TG) and differential scanning calorimetry (DSC) analysis of both the control and the treated jute fabrics were conducted to understand the mechanism of developed flame retardancy in jute fabric. It was observed that the SMSN treated samples showed an excellent antimicrobial property against both the gram positive and the gram negative bacteria. The antimicrobial properties of both the control and the treated jute fabrics were also measured quantitatively.

Keywords: Antimicrobial, Flame retardant, Jute, LOI, Ligno-cellulosic, Sodium Metasilicate nonahydrate,

Introduction

Jute is one of the most important biodegradable, eco-friendly and renewable natural fibre[1]. Apart from its traditional use as a packaging material, jute fabrics are now being widely used in furnishing textiles, upholstery and home textiles[2]. Jute is a ligno-cellulosic fibre, but cellulose being the major component, it catches flame readily, and burns completely in an open atmosphere. As a result, jute based products such as curtains, upholstered furniture, home textiles, and automobile interiors are difficult to extinguish when they catch fire and get quickly damaged[3]. Since the users are more conscious of safety of their life, the demand for flame retardant jute products is steadily increasing. Besides, the demand for durable health and hygienic jute products free from any contamination is also increasing. Therefore, research effort is on to develop dual functional—antimicrobial and flame retardant jute fabrics for home and technical textile application. Different chemical formulations have been reported for flame retardant finishing of jute fabrics for specific end uses [4, 6]. It has been found that phosphorous based flame retardant along with nitrogenous compound work in synergism and is the most effective formulation reported so far [4]. However, jute fabric treated with such chemicals adversely affects the mechanical properties of the fabric, as the finish is applied in acidic condition. Also the treatment process is quite lengthy and expensive due to the involvement of a large quantity of chemical and high curing temperature[5]. Besides, many of the reported technologies are non-durable to washing[6]. Treatment of wood with silicate for flame retardancy has been reported[7]. Other ligno-cellulosic, non-textile materials like lumber, plywood, particle board and wafer board, also have been treated by sodium silicate (not with SMSN) and their flame retardant properties have been evaluated [8, 9]. Till date, use of Sodium Metasilicate nonahydrate (SMSN) for flame retardant and antimicrobial finishing of any textile fabric has not been reported to the best of our knowledge. In this study, the application of SMSN for imparting dual functionality such as flame retardant and antimicrobial property on jute fabrics have been reported. It is expected that such process would also reduce the cost and time of processing and the effluent load, as only one chemical will be used to provide both the functionalities. The flame retardant properties of the control and the treated fabrics were examined with a vertical flammability tester, and by

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determination of LOI value. The mechanism of flame retardants, so imparted to the fabric has been substantiated by thermo gravimetric analysis (TGA) and differential scanning calorimetry (DSC) study. The antimicrobial property of the control and the treated fabrics were evaluated by a quantitative method using the most commonly used gram positive and gram negative bacteria.

Materials and methods

Material

A plain woven jute fabric of 350 g areal density consisting of 12 threads in horizontal and 13 threads in vertical direction was procured. The sodium Metasilicate nonahydrate (SMSN), commonly known as "water glass" was supplied by Qualigens Fines Chemicals, Mumbai. The jute fabric was submerged in water (1: 2) at room temperature for 3 h initially, then dried and conditioned at atmospheric condition for 12 h. The fabrics were then padded with aqueous 2, 4, 6, and 8% (W/V) solution of SMSN (Na_2SiO_3, $9H_2O$) at the room temperature and then dried at 110°C for 5 min. The fabric samples were conditioned in standard atmosphere i.e., at 27°C and 65% RH for 48 h prior to characterisation.

Thermal characterization of the fabrics

The vertical flammability of the control and the treated samples were measured as per IS 11871 method A, in terms of flame time, after glow time, burning rate and char length. For LOI analysis, ISO 1501 test method was used. The Thermo Gravimetric Analysis (TGA) of the control and the treated fabrics were carried out using TG Analyser (METTLER TOLEDO TG-50/ MT5) over the temperature range of 50 to 800°C in N_2 atmosphere with heating rate of 10°C/min. It measures the thermal stability of the fabric [10].The DSC of the control and the SMSN treated jute fabric were carried out using a METTLER TOLEDO DSC-30 thermal analyzer. The heating rate was maintained at 10°C/min in an N_2 atmosphere.

Physical and chemical characterization

(i) Determination of add-on%

In all the treated fabrics, the add-on percentage was determined gravimetrically by measuring the bone dry weight of the sample before and after the treatment, and expressing the results as a percentage of the initial bone dry weight of the sample as follows.

Add-on % = $[(M_2-M_1) / M_1] \times 100$

Where, M_1 and M_2 are the bone dry weights of the control and the treated fabric samples, respectively. The reported results are average of five readings.

(ii) Antimicrobial assay

The different concentrations of SMSN solution were screened for antibacterial activity by an agar well diffusion method with a cork borer of size 9 mm. A bacteria overnight broth cultures grown at 37°C were used in the study. An aliquot (0.1 ml) of tenfold diluted inoculum was seeded into molten and cooled to 45°C nutrient agar buffs and overlaid on sterile nutrient agar. The wells were then punched with exactly 0.1 ml of 2, 4, 6, and 8% of the SMSN solution and introduced into the wells, allowing 10 min for diffusion at 4°C, followed by an incubation period of 24 h at 37°C. The antibacterial activity was evaluated by measuring the size of the zone of inhibition of bacterial growth around the well. For quantitative analysis, both the control and the SMSN treated jute fabrics, the percentage reduction was calculated as per the AATCC-100-2004 standard method.

Reduction in the number of colonies (%) = $[(A-B) /A] \times 100$

Where, A is the number of bacteria recovered from the inoculated treated test specimen swatches immediately after the inoculation at "0"contact time, and B is the number of bacteria recovered from the inoculated treated test specimen swatches incubated over the desired contact period. In the study, *Staphylococcus aureus* (Sa) (Gram positive) and *Klebsiella pneumoniae* (Kp) (Gram negative) bacteria were used.

Results and Discussion

LOI and vertical flammability

Jute fabrics were treated with different concentration of SMSN, such as 2, 4, 6 and 8%. The results of the flammability test and LOI of both the control as well as the treated fabric have been presented in Table 1.

*Sample is self extinguishable after 30s of afterglow

**Total burning time of the fabric= combustion with flame time + combustion with afterglow (after the flame stopped)

It can be seen from the Table 1 that the LOI value of the control jute fabric is 21, which could burn easily in the open atmosphere. However, in all the treated jute

Table 1—Flammability properties of control and SMSN treated jute fabrics

Flammability parameters	Control	SMSN TreatedConcentration (%)			
		2	4	6	8
Add-on (%)	0	1.6	3.5	4.8	7
LOI	21	29	32	37	43
Vertical flammability					
Status of fabric in contact of flame	Completely burnt (with flame)	Completely burnt (initially with flame followed by 600s afterglow)	Completely burnt (initially with flame followed by 1800s afterglow)	Completely burnt (2400s afterglow only)	Very few burning (with afterglow only for 30 s*)
Burning with flame time (s)	60	10	5	Nil	Nil
Total burning time (flame time+ afterglow time)	60s+0	10s+600s	5+1800s	0+2400s	0+30s**
Char length (mm)	-	-	-	-	7
Burning rate (mm/min)	250	25	8.3	6.2	(*)

fabrics, the LOI value increases linearly with the increasing SMSN add-on% on the fabric. It increased from 21 in the control sample to 29 (almost 1.5 times) in the 2% SMSN treated sample. At 8% treated sample the LOI value increased to 43, which is almost double than that noted with the control fabric. In the case of the vertical flammability of the control fabric, the flame spread very fast and burnt the entire sample of (4 x 25) cm^2 within 60s, whereas in the case of only 2% SMSN treated sample, the fabric showed a flame time of 10s followed by its complete burning with afterglow in the 600s. In all the treated fabrics, as the LOI value increased with the increasing add-on of SMSN, the afterglow times were also found to increase to 600, 1800 and 2400s in the 2, 4and 6% SMSN application on the treated samples, respectively. This is an indication of reduction of burning rate of the sample, as the size of the sample is constant. The burning rate reduced from 250 to 25mm/min in the control to the 2% SMSN treated samples. It further decreased with the increasing percentage application of the SMSN. Ultimately, the 8% treated sample does not catch flame, however, the sample burns partially for only 30s with afterglow, and then get self extinguished producing 7 mm char length only for this particular sample. The durability of the imparted SMSN finish to soap washing was studied to evaluate its long term performance in some applications. The 8% SMSN treated jute fabric was washed as per the ISO 2 washing method. It was observed that after the soap washing, the LOI value decreased from 43 in the as-prepared sample to 30 in the washed sample, which is almost equal to the 2% SMSN treated sample. Hence, it can be said that the SMSN treatment could be used to impart semi-durable flame retardant finishing. It might be due to the electrostatic inter-attraction and complex formation of SMSN with hydroxyl groups of the jute cellulose.

Thermogravimetry

It can be seen that the Thermo gravimetric (TG) curve of the pure SMSN showed a 60 % mass loss in the range of 50 to 200°C. This is probably due to the loss of unbound and bound water molecules in different ways[13]. However, after 200°C, there was no significant change in mass till 800°C. Compared to control jute fabric (A), the SMSN treated jute fabric (B & C) loses more mass below 150°C. It might be attributed to the loss of nine molecules of water of the SMSN in addition to loss of water molecules of jute fabric [14-16]. In the second stage, both the 4% and 8% treated jute fabrics started to lose its mass due to pyrolysis from 230°C, which is quite below by about 35°C than the pyrolysis temperature 265°C of the control fabric. From the observation, it can be deduced that the presence of SMSN in the jute fabric, reduces the pyrolysis temperature of the jute polymer and increases the overall resistance of the treated fabric to thermal degradation. The same also might be attributed to the dilution of flammable, volatile gases by the generation of non-oxidizable CO_2, and H_2O at comparatively lower temperature[11] .In the 8% SMSN treated jute fabric (C), the char formation started at 325°C, at a slightly lower temperature than the control sample, and the residual mass increased from 10 to 22% at 500°C.

Differential Scanning Calorimetry (DSC) analysis

Differential Scanning Calorimetry (DSC) analysis was carried out for both the control and the SMSN treated jute fabric to determine the thermal behaviour of the fabrics as shown in Fig. 2. An endothermic peak was observed in the temperature range of 50-130°C in both the control and the SMSN treated jute fabrics corresponding to the heat of evaporation of water molecules absorbed in the fabric[15].The observed endotherm was found to be steeped in the case of the treated fabrics. This might be attributed to the vaporization of nine molecules of water of SMSN from the treated jute fabric in addition to removal of absorbed moisture from the jute fabric. This is because jute could absorb 13% moisture of its own weight. For the control jute fabric (A), the DSC thermogram showed an endothermic minimum at around 380°C. This endotherm corresponds to the depolymerisation of cellulose with the formation of flammable gases like levoglucosan[15]. At this temperature, the pyrolytic degradation took place with rapid cleavage of the glucosidic bond[15]. Whereas, the DSC thermogram of the treated jute fabrics (B & C) revealed that the endothermic peak has reduced to a small endothermic bend at lower temperature (B) or has been almost disappeared (C). Therefore, like in the TG analysis, it can be interpreted that the SMSN treatment dilutes the formation of flammable gases, as a result of which the LOI values of the treated fabrics increase and the flame time and burning rate decrease. Another interesting observation from the DSC thermogram of the treated jute fabric is the appearance of an exothermic peak at 310°C (C) for the 8% treated sample and at 340°C (B) for the 4% treated sample. This exothermic peak is responsible for dehydration of cellulose polymer and char formation[16, 17]. A similar phenomenon happened in slightly higher temperature in the control sample (A). This implies that the exothermic peak, which is related to the dehydration of hemi-cellulose and

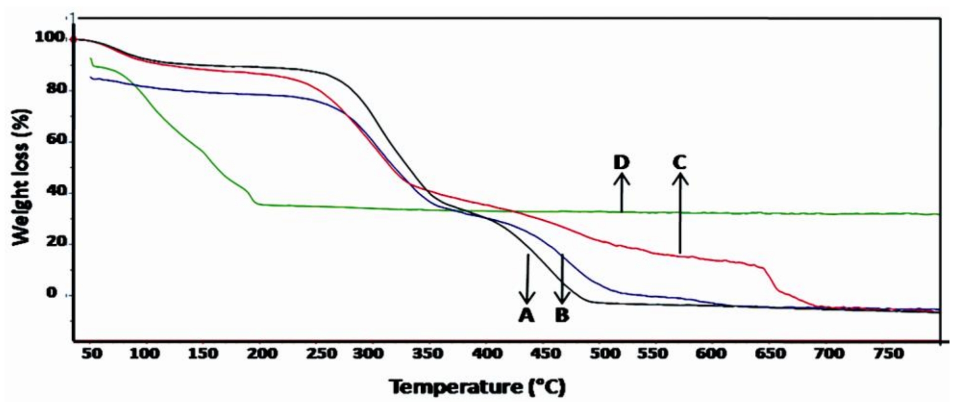

Fig. 1—TGA curve of control jute (A), 4% SMSN treated jute fabric (B), 8% SMSN treated jute fabric (C) and pure SMSN (D)

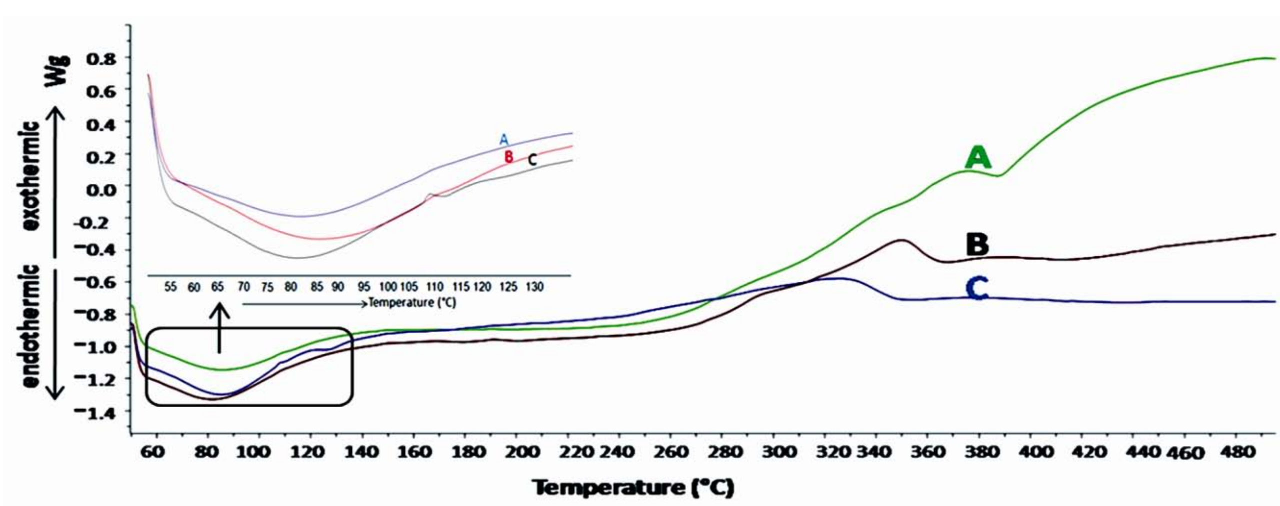

Fig. 2 —DSC curves of control (A), 4% SMSN treated (B) and 8% SMSN treated (C) jute fabric

cellulose, has shifted to lower temperature from 340 to 310°C, when the SMSN add-on is increased from 4 to 8%. Thus the presence of SMSN helped in lowering down the degradation temperature of the treated jute fabric and production of more non-flammable gases and char. Besides, the area under the exothermic peak was also found to increase with the increase in SMSN content. Therefore, like in the case of TG analysis, this phenomena could be attributable to the dehydration of cellulose that occurred more extensively as the SMSN content in the fabric was increased.

Mechanism of flame retardant property

The application of SMSN possibly forms a silicate coating on the surface of the jute fabric that which acts as an intumescent for the non-textile substrates[7]. It serves as a hydrated plaster. Upon exposure to high temperature, it swells and increases in volume, but decreases in density. It also forms an insulating carbonaceous foamy glassy layer on the surface of the treated fabric[7, 10, 12]. It has been reported that insulating layer contains char producing compounds such as, polyol, which normally burns to produce non-oxidizable gases like CO, H_2O and CO_2. These generated gases might have diluted the flammable gases such as levoglucosan and pyroglucosan produced during pyrolysis of jute samples in the burning microclimate[7, 11]. Further, the chemically bound nine molecules of water with the SMSN absorb a significant amount of heat during the burning process, and thus, reducing the propagation of fire.

Antimicrobial assay

From the screening results of the different concentration of SMSN solutions, it was evidence that the SMSN solutions had inhibited both the gram positive and the gram negative bacteria. It was observed the antibacterial activity was increased gradually with the increasing in the percentage concentration of SMSN. Thus, at 8% concentration, the diameter of the zone of inhibition was found 18 mm and 17 mm against *Staphylococcus aureus* (Sa) and *Klebsiella pneumoniae* (Kp) bacteria, respectively. The quantitative evaluation of anti-bacterial activity was carried out on both the control and the SMSN treated fabrics against *S. aureus* and *K. pneumoniae* as per the AATCC-100-2004 standard method. A typical photograph of bacterial reduction is shown in Fig. 3. It can be seen from the experiment that 2% SMSN treated fabric has 62.2% and 75.5% potential to reduce the colony growth against Sa and Kp bacteria respectively. It was increased linearly with the percentage application of SMSN. Thus the reduction in bacterial growth was 99.9% in the 8% treated jute fabric, which is considered to be an excellent antibacterial textile material. However, from the result, it was seen that the SMSN treatment is more effective against gram negative (Kp) bacteria compared to gram positive (Sa) bacteria. The antibacterial activity of the treated fabric might be attributed to a higher pH value of the SMSN[18], and also due to the breakage of membrane integrity and disruption in the cytoplasmic membrane of the bacterial wall in presence of SMSN. A similar phenomenon has also been reported earlier in the literature[19-21].

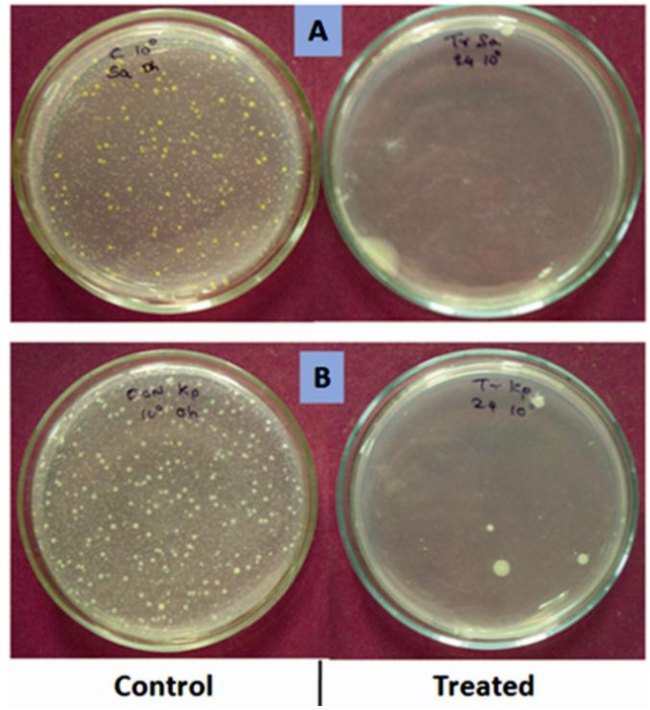

Fig. 3—Bacterial reduction in 8% SMSN treated jute fabric against (A) *Staphylococcus aureus (Sa)* and (B) *Klebsiella pneumonia (Kp)* bacteria compared to the control sample

Conclusion

The present study showed that both the flame retardancy and the antimicrobial properties of SMSN treated jute fabrics were quite good, and the 8% formulation was found to be superior for the application by padding method. If the fabric is meant for application like, interior material for passenger cars, trucks, buses, and for making decorative home furnishing materials like table lamp, wall mat, curtains etc., then this technology will be useful for imparting both the fire retardancy and antimicrobial property in the textiles. Since SMSN is odourless, non toxic, abundantly available, inexpensive and the reported process is simple, the developed technology will be useful to impart dual functionalities i.e., flame retardancy and antimicrobial property in jute textile effectively and economically.

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