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Flame retardant characteristics of polymerized dopamine hydrochloride coated jute fabric and jute fabric composites

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Abstract: In this paper, fire resistance of natural fabrics and their composites were experimentally investigated. Special interest was given to use bio based materials such as lignin, chlorophosphates, levulinic acid and cardanol in order to exploit their capability to be utilized as flame retardants. Dopamine hydrochloride was polymerized to polydopamine (PDA) and coated to jute fabric surface. Scanning electron microscope (SEM) and thermogravimetric analysis (TGA)/derivative thermogravimetric (DTG) analyses were performed to examine surface morphology and effect of PDA to degradation behaviour of jute fabrics. Real fire behaviour of non-coated and coated fabrics was observed with torch burn test. UL-94 horizontal flame propagation test was also utilized for composite samples. Limiting oxygen index (*LOI*) testing that measures the minimum amount of oxygen required for combustion, was carried out for assessing the ability of the composite samples for their ability against flammability. PDA was seamlessly coated on the surface of the jute fabrics with its surface-active feature without damaging the structure of the fabric as observed in the SEM images. With the support of this coating on the fabric surface, the increase of the decomposition temperature of the material can be clearly seen in TGA/DTG analyses and torch burn test showed the increase in the ignition time. UL-94 horizontal testing resulted in decrease in flame propagation rate of PDA coated composite samples. In addition to this, when the mass loss rates after combustion were examined, it was seen that there is a decrease in mass loss in the coated fabrics. Jute fabrics, a type of natural fabric, can be efficiently coated with PDA, and the fire retardant property of the PDA coating on natural fabrics has been clearly demonstrated.

Keywords: fire resistance; organic coatings; natural fibre composites.

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INTRODUCTION

Natural fibres are fibres originated from natural resources, *i.e.*, animals or plants.[1] The use of natural fibres as the reinforcing phase for the composites has become attractive in the recent years because of their low cost, friendly processing, renewability and biodegradability features.[2] Although having many advantages of using natural fibres in the composites, there are some disadvantages of these fibres, *e.g.*, mechanical, thermal and physical. In the literature more attention is given to the mechanical and physical performance of these fibres than to fire characteristics of natural fibres and their composites.[3,4]

Like many natural phenomena, the first phase of fire process starts on the surface by ignition. So it is of the utmost importance to concentrate on the interactions happening on the material surface. One of the surface modification methods is coating, and the purpose for coating is to modify a surface to meet the desired requirements by adding a new layer of material or compound. The use of several types of coating has been reported as an effective approach to improve the fire safety of the materials where the thin layers of less than 100 nm could be achieved.[5]

Coatings used as fire retardants protect materials by delaying the ignition time. The applications of the fire retardant coatings are one of the most efficient and easiest methods without making modifications in the intrinsic properties of the host material.[6] Several types of coatings such as magnesium hydroxide, ammonium polyphosphate, and graphene have been utilized as fire retardants including metallic, organic and inorganic substances respectively.[7] Recently, organic and toxic free fire retardants are gaining attraction because of health issues.[8] Baldissera *et al.*[9] have studied semiconducting polymer as fire retardant on mild steel surfaces. They found out that the coating has improved both anticorrosive and fire protection capabilities of the coated structure. Hybrid organic and inorganic fire retardants for the use in fabrics have been studied by several researchers.[10–12] In these studies reported success was the synergistic effect of the organic-inorganic compounds improving the flame retardancy and self-extinguishing effects.

Polydopamine (PDA) is a bio based mussel inspired polymer that was discovered in 2007 by Lee and colleagues.[13] Because of the presence of PDA inside the mussels they have the ability to strongly adhere to the surfaces of other substances. It is reported that as PDA has a similar structure with mussels, and it can easily be coated on any type of organic or inorganic substances.[14]

PDA can be used as coating almost on any material where coating thickness of few to one hundred nanometres could be made.[15] As nontoxic and environment friendly material, PDA could be employed as flame retardant coating which contains catechol molecule. In a study made by Cho *et al.*[16] polyurethane (PU) foam was deposited by nanocoatings of PDA to see the effects of PDA thickness on the flame properties. It was seen that flammability of the PU material was reduced with the increasing PDA thickness. After 3 days of PDA deposition, torch burn experiment was carried out on the sample and it was seen that PU material quickly self-extinguished and preserved its original shape.

Li *et al.*[17] studied flame retardant and antibacterial properties of cotton fabrics coated by hybrid cyclotriphosphazene, PDA and silver nanoparticles. They found out a coating yield of 3.7, 7.2 and 16.9 % by coatings with different flame retardant concentrations such as 10, 30 and 60 g/L on the fabrics. Flame retardancy was considerably improved and excellent antibacterial activities against *Staphylococcus aureus* and *Escherichia coli* were obtained.

Several researchers have reported the studies for flame retardancy and fire phenomena of natural fibre composites.[18–20] Major chemical constituent of jute is cellulose but it also contains other components such as lignin, hemicellulose and inorganic salts often identified as ash.[21] These non-cellulosic components play an important role in the properties of jute. For example, lignin, a high molecular weight polymer based on phenyl propene, provides cell wall stiffness.[22,23] Jute, bast type of fibre, is one of the most commonly used natural fibres and traditional areas of use for the jute fibre are bag, rope and bed production. Jute fibre based natural composites are used in some structural applications such as indoor elements in housing, low cost housing for defense, rehabilitation and transportation. Jute fibres have good insulating properties and the produced parts are used in door/ceiling panels.[24] Saleem *at al.*[25] manufactured jute reinforced mud bricks by using a compression machine to improve the compressive strength of the samples. They found that after 28 days of drying under sun, compressive strength of jute reinforced mud bricks improved up to 2.75 times compared to that of mud bricks without fibres. Although jute and other natural fibres could be used as reinforcing material in the chopped form, it gives more strength when they are used in woven form.[26] By this way, non-directional and randomly distribution disadvantages of the chopped fibres disappear.

Like synthetic fibres, polymers are utilized to form a composite material and this kind of material is called polymer matrix composite. It is reported in the literature that many thermoset and thermoplastic polymers, either bio based or not, are compatible with natural fibres for the composites production.[27,28] As the operating temperature of natural fiber composites increases, its strength and stiffness decrease. In the elevated temperatures or when exposed to flame, natural fibre composites experience thermal decomposition and combustion. Combustibility of natural fibre composites depends on the nature of its constituents, thermal conductivity, density, structure, humidity and *etc.*[20] Polymeric resins are attractive in the natural fibre composites manufacturing due to ease of processing, relatively low cost and suitability to use. However, the utilization of different polymeric resin brings some problems as well too; these hydrocarbon based materials can be flammable and even toxic and make contribution to flame propagation when in contact with a heat source.[29] As was mentioned before, jute like other natural fibres are prone to fire and has low fire resistance. When combining with the polymeric resin to form a composite material, fire resistance of natural fiber composites becomes even worse.

This paper presents the development of fire resistant jute fabric and its composites by PDA coating on the jute fabric. The PDA was obtained by polymerizing dopamine hydrochloride. Reference uncoated jute fabrics and uncoated jute fabric composites were also produced for comparison purposes with their PDA coated jute fabrics and PDA coated jute fabric composite counterparts. Torch burn testing as well as UL-94 horizontal tests were carried out on the samples and SEM, TGA and DTG analysis were also performed. Additionally, *LOI* testing was carried out to observe resistance of the PDA coated composite samples against flammability. The main aim of this study was to produce and test organic and nontoxic coating on a natural fabric, this was achieved by efficient coating with PDA, and fire retardant property of the PDA coated jute fabrics has been demonstrated.

EXPERIMENTAL

PDA coating on jute fabrics

One group of fabrics was used as it is (uncoated), while the other group (for contribution to combustion resistance) was coated with PDA. 100 ml of Tris-HCl buffer solution was prepared with a pH of 8.5. First, dopamine hydrochloride was added to the solution. Dopamine hydrochloride (3-hydroxytyramine hydrochloride) was obtained from Sigma–Aldrich. Subsequently, jute samples were immersed in 1 mg/ml PDA solution and mixed with magnetic stirrer for 12 h at room temperature. Several jute fabrics were (PDA coated and uncoated) utilized for TGA/DTG and SEM analysis while the remaining uncoated and PDA coated fabrics were stored in a shelf with dehumidifier for the composite plate production. The jute fabrics that were used in this study are tight woven 250 g/m^2. The mass of each 5×5 cm^2 jute fabric used to produce the composite plate is approximately 0.625 g. The dimensions were measured by a digital calliper and the masses by a precision balance.

Fabrication of composite plates

Jute fabric epoxy composites were prepared by hand lay-up method of 5×5 cm^2 jute fabrics. In these processes, after the epoxy was applied on the fabrics homogeneously with a brush, the next jute fabric layer was placed on this layer regardless of the weaving direction, and in this way, the epoxy impregnation process continues for 5 layers. The epoxy resin used in this study was obtained from Duratek Company and Duratek 1200 + 2110 epoxy system was used as matrix of the composite.

Hot pressing technique is a widely used manufacturing technique for the production of such composite plates. In this production technique, the temperature and pressure are applied to the sample placed between two flat metal plates. In our study, jute fabrics impregnated with epoxy resin by hand laying method were kept at 100 °C under 0.4 tons pressure for 4 h and turned into a composite plate. In Fig. 1, PDA coating process on jute fabrics and composite plate production phase are given schematically.

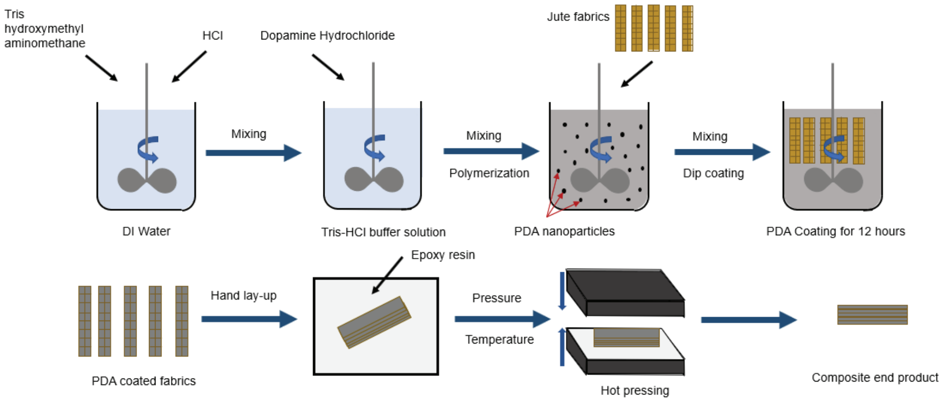


Fig. 1. Schematic representation of PDA coating and composite plate production.

Characterization

Characterization methods were applied to examine the coating on the material surface and the performance of this coating in accordance with the purpose of the study. In this context, SEM (Hitachi SU5000) analyses were performed to examine the layer formed by the PDA coating on the jute fiber surface. Thus, these images gave us information about the surface morphology of PDA coated jute fibers. SEM images of several composite plates were obtained after UL-94 testing.

TGA/DTG (Hitachi STA 7300) analyses were conducted to obtain information about the degradation temperatures and the change in their mass with temperature increase of PDA coated and non-coated jute fibre samples upon 600 °C under N_2 atmosphere.

Finally, the flame behaviour of composite materials and coated jute fabrics was examined with a torch burn test apparatus. With this examination, information about the ignition times of the materials and flame propagation comparisons and the mass loss rates before and after the combustion were obtained.

Torch burn tests

In order to examine the fire retardant effect on PDA coated jute fabrics, two types of samples produced were exposed to flame source at equal distance. For this purpose, a butane torch which provided a blue flame was utilized and the behaviour of the samples (*i.e.*, ignition time and flame propagation time) was analysed visually. This test unlike UL-94 horizontal testing does not give any quantitative data on the propagation rate of the flame, but instead gives qualitative information on the propagation rate of the flame. The torch burn test gave the ignition times of both jute fabrics and its composites.

Underwriters laboratories – 94 (UL-94) horizontal tests

A cabinet was designed and built by the authors for the purpose of performing UL-94 testing. A total of six jute fabric composite samples have been prepared and put to UL-94 horizontal testing for the assessment of the samples' flame propagation behaviour in accordance with ASTM D635. Table I shows the geometrical properties of the samples used in the UL-94 horizontal testing.

After producing six samples, based on the dimensions given in the Table I, a vertical line indicated as L_i was drawn 75 mm away from the edge of each sample with the help of a permanent marker as shown in Fig. 2.

TABLE I. Geometrical properties of samples used in the UL-94 testing

Sample	Sample No.	Thickness, mm	*L* / mm	*W* / mm	No. of layers
Uncoated jute-epoxy composite	1, 2, 3	2.5	130	10	5
PDA coated jute-epoxy composite	4, 5, 6	2.5	130	10	5

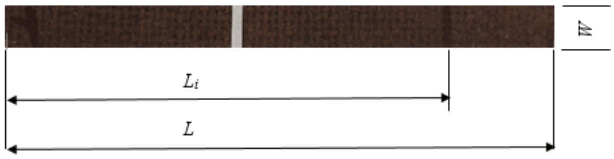


Fig. 2. Dimensions of sample used for the UL-94 testing.

*Limiting oxygen index testing (*LOI*)*

LOI testing is one of the efficient ways to assess the flammability of the materials, by doing this the minimum amount of O_2 needed to burn the material was investigated based on ISO 4589. During the testing, the amount of O_2 required for combustion of PDA coated composite samples (samples 10–12) compared to uncoated composite samples (samples 7–9) was examined. *LOI* testing of composite samples (coated and uncoated composite plates) was carried out with a dimension of 80×10×2.5 mm^3. The results are given in results and discussion.

RESULTS AND DISCUSSION

The change in the colour of the fabrics from yellow to dark brown clearly demonstrated the successful coating of the PDA in Fig. 3.

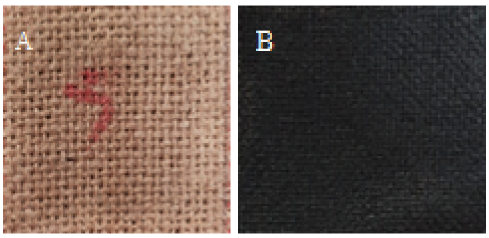


Fig. 3. Photographs of the prepared samples, A) before PDA coating and B) PDA coated fabrics.

The SEM images of the PDA coated jute fabrics are presented in Fig. 4. As seen here, a uniform PDA growth was achieved with no significant change in the fibre structure of the jute fabrics. Additionally, further magnification on the surface of the PDA coated jute fabrics demonstrated the conformal coating of the PDA nanoparticles with no change in the uniform morphology of the fibres (Fig. 4E and F). Moreover, large PDA grains, which were clearly shown in SEM images, demonstrated the strong adhesion of the PDA from bulk to the surface of jute fibres (Fig. 4G and H).

SEM images of PDA coated composite jute plates after UL-94 horizontal testing (Fig. 5A and B) show evidence of good adhesion of the PDA even after the fire has been extinguished. As seen in Fig. 5A, PDA has formed a protective char layer on the fibre surface. For this reason, during the combustion the heat penetrated less into the interior of the jute fabrics and the fabric material, maintaining its integrity, did not act as a combustible material. In addition, this char layer restricts the passage of flammable gases to the combustion zone even if pyrolysis occurs in the material inside and prevents the strengthening of the flame. Figure 5B shows the distribution of the char layer formed by PDA on the fibres after combustion.

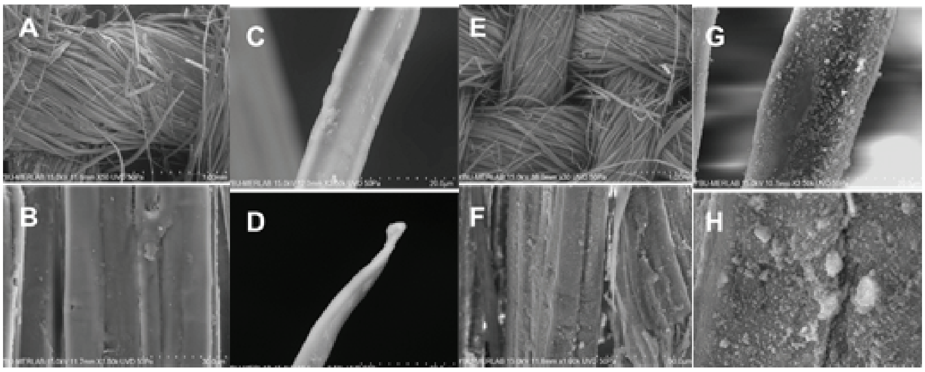
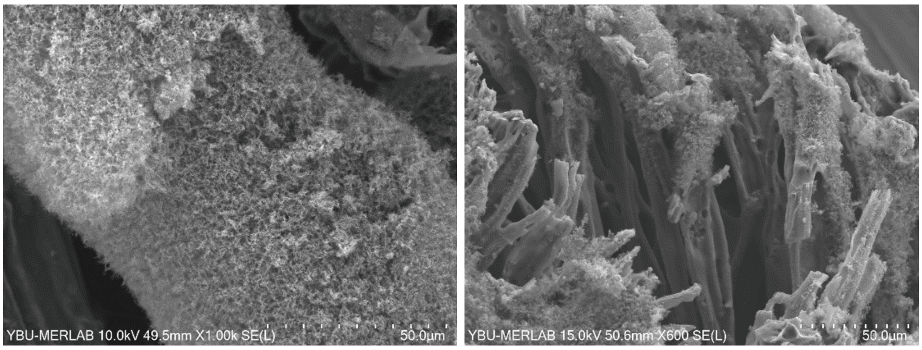


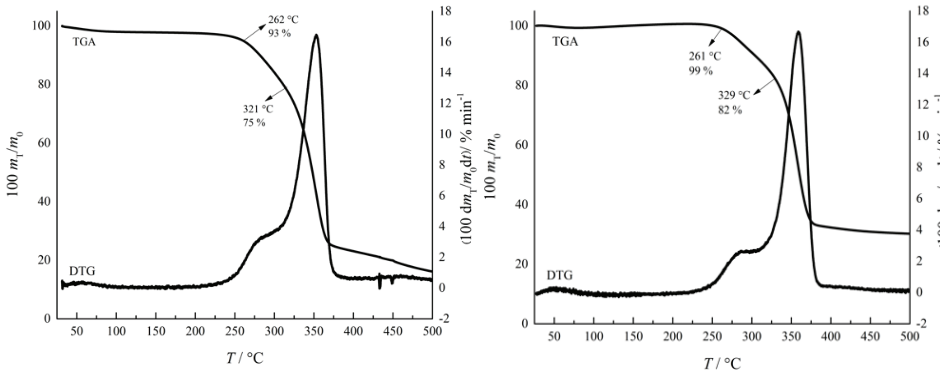
Fig. 4. SEM images of: A)–D) jute fabrics before PDA coating and E)–H) PDA coated jute fabrics.



(A) (B)

Fig. 5. SEM images of PDA coated composite jute plates and char formation after UL-94 horizontal testing: A) PDA coated single jute fiber char formation after combustion; B) Distribution of char layer on the PDA coated jute fiber after combustion.

In Fig. 6 as can be seen, the TGA curves show two clear mass loss events in the structure of bare jute and PDA coated fabrics. The first mass loss of 6.1 and 1.33 %, in the range of 200 to 320 °C, was measured for bare jute and PDA coated jute samples, respectively. From the DTG curves (Fig. 6A and B), it could be noted that the bare jute and PDA coated jute samples decomposed at 310 and 365 °C for bare jute and PDA coated jute samples, respectively. This phenomenon showed the high amount of mass loss at approximately 310–400 °C for second stage. Hong *et al.* examined the thermal properties of bamboo fibres by coating PDA, and proved that the degradation temperature of PDA coated bamboo fibres increased compared to the uncoated fibres.[30] Moreover, the shifting in the DTG curve of the samples could be caused by the reactive surface of the PDA nanoparticles (*i.e.*, radical scavenging), presences of catecholic groups, as well as reducing the heat release from PDA coated jute samples.[31]



(A) (B)

Fig. 6. TGA/DTG analysis of: A) pristine jute fabric and B) PDA coated jute fabric.

Flame retardant behavior

Reference single layer jute and reference composite jute ignited instantly and the flame propagation rate of single layer jute is faster than PDA coated single layer and flame propagation rate of reference composite jute is faster than PDA coated composite jute samples. As shown in the Table II, the ignition times of the PDA coated samples are considerably higher than that of the reference samples. Unlike the reference samples, the flame did not directly contact the surface of the samples, firstly come into contact with PDA coating on the surface.

TABLE II. Some parameters related to the torch burn tests and flame retardant behaviour of samples

Material type	Composition	Ignition time, s	Flame propagation rate comparison	Mass loss rate wt.% s^{-1}
Reference jute	Single layer pure	4	Faster than single layer coated	2.87
PDA coated jute	Single layer coated	10	Lower than reference jute	2.49
Reference composite jute plate	5 layer coated with epoxy	8	Faster than 5 layer coated with epoxy	0.93
PDA coated composite jute plate	5 layer coated with epoxy	25	Lower than 5 layer with epoxy	0.47

Besides increasing the ignition time, low flame propagation rate is a significant point during fire. Fire tests confirmed that PDA coating decreased flame propagation rate. Fox *et al.* in the flame propagation test of the polyurethane foam and cardboard coated by the PDA, showed that the PDA coating eliminated the flame propagation.[32] While the flame propagated on the reference samples rapidly, it showed more slowly propagation behavior on the PDA coated samples. Taking these two results into consideration, the PDA coating clearly demonstrated the flame retardant effect by delaying ignition and reducing flame spread rate. In addition to these results, as seen in the table, mass loss rates have been reduced significantly.

UL-94 horizontal testing was performed inside the cabinet designed and built by the authors. Fig. 7A and B shows the stages of UL-94 horizontal testing of a PDA coated composite sample.

Fig. 7. PDA coated composite plate: A) ignition phase and B) propagation of the flame.

In Fig. 8, the chart showing comparison between uncoated and PDA coated jute-epoxy composite samples' flame propagation rate has been given. In this figure, time to extinguish has been given for a total of six specimens. Three PDA coated jute–epoxy composite and three uncoated jute-epoxy composite plates were put on testing. Time to extinguish resulted 366, 338 and 355 s for PDA coated samples number 4, 5 and 6, respectively, while time to extinguish gave 140, 177 and 101 s for uncoated samples number 1, 2 and 3, respectively. The arithmetic average value of uncoated samples time to extinguish value is calcul-

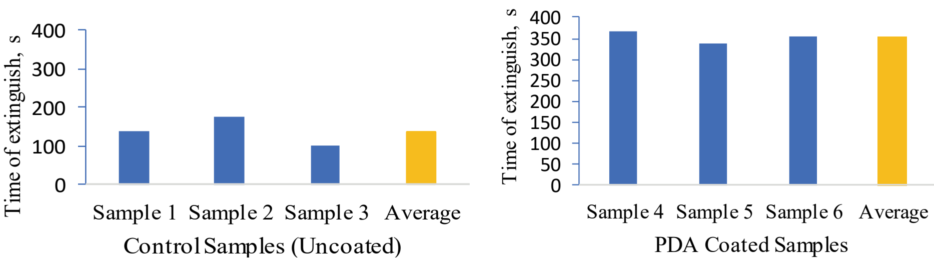


Fig. 8. UL-94 horizontal testing of composite plates.

ated as 139 s and coefficient of variation is 27.33 % while the arithmetic average value of PDA coated samples is calculated as 353 s and coefficient of variation is 3.97 %. The standard deviation of uncoated jute–epoxy composites is 38, while the standard deviation of PDA coated samples is 14.10. When arithmetic average values are compared, it is seen that time to extinguish values of PDA coated samples' flame propagation rate are about 60 % less than that of uncoated samples. UL-94 horizontal testing proved the efficiency of PDA coating on jute fabrics with regard to flame propagation.

LOI *results*

As given in Fig. 9, uncoated composite samples are the most combustible with 19 % limit amount of O_2 during the testing. PDA coated composite samples with 23 % O_2 content started burning showing better combustion performance than their uncoated counterparts.

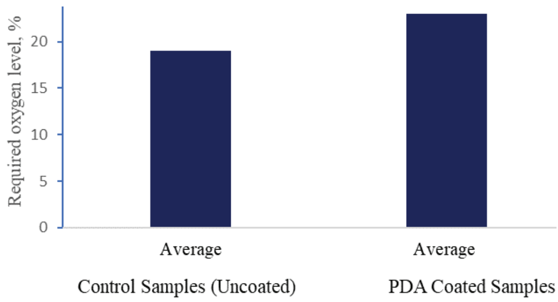


Fig. 9. LOI testing of composite plates.

Wang *et al.* observed an increase in *LOI* value when they coated ammonium polyphosphate and PDA to cotton. Although, in their study, the greater increase showed the effect of ammonium polyphosphate, the *LOI* value which was 26.5 %, increased to 28.5 % with the effect of the PDA coating.[33] Zhang *et al.* produced flax–PLA composite with PDA and iron phosphonate coating. In this study, PDA and iron phosphonate showed fire retardant effect and increased the LOI value from 19.1 to 26.1 %.[34] This testing is defined as the minimum oxygen percentage needed to sustain flaming combustion. As this value gets higher, a material's ability to resist flammability is said to be improved. Compared with uncoated samples, PDA coated samples have improved resistance against flammability.

CONCLUSION

In this study, dopamine hydrochloride was polymerized to PDA and coated to jute fabric surface. After the dipping method coating process, SEM and TGA/ /DTG analysis were performed to examine surface morphology and effect of PDA to degradation behaviour of jute fabrics. Additionally, real fire behaviour of non-coated and coated fabrics was observed with torch burn test. *LOI* and UL-94 horizontal testing were also employed for assessing the flame propagation and combustion characteristics of composite plates as well.

As a result of the study, PDA was seamlessly coated on the surface of the jute fabrics with its surface-active feature without damaging the structure of the fibres as observed in the SEM images. With the support of this coating on the fabric surface, the increase of the decomposition temperature of the material can be clearly seen in TGA/DTG analyses and torch burn test showed the increase in the ignition time. In addition to this, when the mass loss rates after combustion were examined, it was seen that there is a decrease in mass loss in the coated fabrics. Jute fabrics, a type of natural fabric, can be efficiently coated with PDA, and the fire retardant property of the PDA coating on natural fabrics has been clearly demonstrated. The results of the UL-94 horizontal test show that flame propagation is significantly slowed down for PDA coated jute fabric composites, while the *LOI* test shows that the amount of oxygen required for the combustion of the PDA coated jute fabric composite material is increased compared to composites made of uncoated jute fabrics. In this case, while PDA coating decreased the flame propagation rate it increased the minimum amount of oxygen required for combustion.

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ИЗВОД

ТКАНИНЕ ОД ЈУТЕ И КОМПОЗИТИ НА БАЗИ ТКАНИНА ОД ЈУТЕ СА СЛОЈЕМ ПОЛИМЕРИЗОВАНИГ ДОПАМИН-ХИДРОХЛОРИДА ПОВЕЋАНЕ ОТПОРНОСТИ НА САГОРЕВАЊЕ

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У овом раду је испитивана отпорност на сагоревање природних тканина и њихових композита. Посебна пажња посвећена је биоактивним материјалима, као што су лигнин, хлорофосфати, левулинска киселина и карданол да би се испитала њихова способност успоравања ширења пламена. Допамин-хидрохлорид је полимеризован у полидопамин (PDA) и нанет на површину тканине од јуте. Скенирајућом електронском микроскопијом (SEM) је испитана морфологија површине, а термогравиметријском анализом (TGA) утицај PDA на деградацију тканине од јуте. Отпорност на сагоревање сирове тканине и тканине наслојене PDA је испитана применом горионика. За композите на бази јуте је коришћен и UL-94 хоризонтални тест горења. Гранични индекс кисеоника (*LOI*) којим се одређује минимална количина кисеоника потребна за сагоревање је примењен за процену отпорности композитних материјала на сагоревање. SEM анализа је показала присуство танког слоја PDA на површини тканине, без нарушавања њене структуре. Захваљујући овом слоју, температура на којој долази до деградације материјала је повишена, што је утврђено TGA анализом, а време сагоревања је продужено, што је утврђено тестом са горионом. UL-94 хоризонтални тест горења је показао смањење брзине ширења пламена за узорке наслојене PDA. Додатно, губитак масе након сагоревања је смањен у случају узорака са слојем. Резултати показују да се тканина од јуте, врста природне тканине, може ефикасно препокрити полидопамином, што доприноси побољшању отпорности на сагоревање.

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