Novel Medium-density Fibreboard Produced by Ultrasonic-assisted Pulp with Superhydrophobic and Flame-Resistant Properties

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A novel medium-density fibreboard produced by an ultrasonic-assisted wheat straw pulp (UWP) was obtained without adhesives. It was then coated with a superhydrophobic sol solution integrated with an *in vitro* addition of two fire retardants (polyhedral methyl-silsesquioxane and ammonium polyphosphate) during the process of sol-gel reaction involving the two silane precursors tetraethyl orthosilicate (TEOS) and tridecafluorooctyltriethoxysilane (FAS). The coated UWP medium-density fibreboard (UPB) had good strength properties and possessed excellent hydrophobicity (water contact angle (WCA) above 150°), and flame-resistant properties (limiting oxygen index (LOI) improved by 5% compared with the original sample with a LOI of 18%). Meanwhile, the fibreboard also exhibited outstanding anti-permeability towards water (kept constant WCA for more than 1 h).

Keywords: Ultrasonic-assisted wheat straw pulp; Medium-density fibreboard; Superhydrophobic sol solution; Flame resistant; Acidic-corrosion resistant

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INTRODUCTION

Currently, the increasing consumption of medium-density fibreboard (MDFB) products has resulted in the increasing demand for woody raw materials. Various non-wood fibres, such as wheat straws, rice straws, corn stalks, and reeds, have been used as alternative materials (Wang *et al.* 2010; Kumar *et al.* 2015; Cao *et al.* 2016; Cutz *et al.* 2016). For example, in China, approximately 300 million tons of rice straws are annually disregarded across the land, 25% of which could be collected and potentially produce at least 75 million m^3 fibreboards (Hu 2004; Li *et al.* 2012). If even a minimal amount of the non-wood materials could be utilized, the phenomenon of an inadequate timber supply would be eased considerably, and the environmental issues that straw burning causes would be solved (Cardoen *et al.* 2015; Hong *et al.* 2016).

However, the drawbacks of traditional wood fibreboard production methods require more attention. One problem is that it is difficult for wood raw materials to produce silk-like fibres *via* conventional methods, which just produce wood chips or wood powders in ugly shapes (El-Kassas and Mourad 2013; Sánchez *et al.* 2016). Another setback is that adhesives are needed to glue the wood chips. The traditional adhesives, such as urea-formaldehyde resin or phenolic resin (Wang *et al.* 2007; Buyuksari *et al.* 2010), result in air pollution due to formaldehyde release. If straws were applied as raw materials instead of wood, it would pose difficulties to obtain satisfactory strength due to the waxy and siliceous character of the straw fiber surface (Van Soest 2006; Zhang and Hu 2014).

To solve the above drawbacks, an ultrasonic-assisted wheat straw pulp (UWP) was considered to produce MDFB, in which the binding force came from the hydrogen bonds between the pulp fibres without using adhesives. Compared to the conventional pulp currently on the market, UWP has lower output costs and better strength properties (Xing *et al*. 2017). Meanwhile, fabricating superhydrophobic and flame-resistant surfaces of medium-density fibreboard also form an important link to overcome the drawbacks of the hydrogen bonds, which easily lose efficacy when contacted with water. In some special cases, superhydrophobic surfaces with flame-resistant properties are required, to be exact, a water contact angle (WCA) above 150° and a limiting oxygen index (LOI) that exceeds 21% (Ravadits *et al*. 2001; Weil and Levchik 2008). Conventionally, the fire retardants are mixed with the hydrophobic polymers to prepare waterproof coatings with flame-resistant properties. However, most of the approaches have involved tedious and multiple-step procedures that are impractical and high-cost (Hikita *et al.* 2005).

As a replacement, the authors adopted a simple and feasible method: a sol-gel method (Guo *et al.* 2011) to prepare the flame-resistant superhydrophobic sol solution, which was obtained by a sol-gel process of the reaction of tetraethylorthosilicate (TEOS) and tridecafluorooctyltriethoxysilane (FAS). Notably, FAS possesses a low surface energy, an excellent durability to acid and is commonly used to provide hydrophobicity of the surface (Xu *et al.* 2008).

With respect to the choice of fire retardants, two factors were considered. First, it was important to determine whether the fire retardants posed a detrimental impact on the superhydrophobic properties. Second, the authors determined whether the fire retardants were environmentally friendly. Because the silsesquioxane-based materials possess a low density, high temperature resistance, and are fire-retarding, the polyhedral oligomeric silsesquioxane (POSS) has been extensively employed as a water-repellency reagent (Tuteja *et al.* 2008) and fire retardant (Dasari *et al.* 2013). For example, Pan *et al.* (2013) prepared a super hydrophobic coating by employing an electrospun solution of cross-linked polydimethylsiloxane (PDMS) and 50 wt.% fluorodecyl polyhedral oligomeric silsequioxane (F-POSS). Li *et al.* (2001) fabricated a super hydrophobic surface on a cellulose-based material, utilizing polyhedral methyl-silsesquioxane (methyl-POSS) to provide hydrophobicity.

Typically, as a fire retardant, the methyl-POSS is mixed with other polymer resins to improve the fire retardancy of the original materials. However, considering the limited fire-resistant efficiency of POSS, a highly effective fire-retardant ammonium polyphosphate (APP) was also employed.

The APP serves as a fire retardant with many unparalleled advantages: (1) excellent fire resistance properties originated from high phosphorus/nitrogen (P/N) content with a P-N synergistic effect; (2) high decomposition temperature and thermos stability; (3) low solubility and hygroscopicity; (4) good dispensability; and (5) low price. Dasari *et al.* (2013) investigated the effect of APP on carbonification and inflaming retarding of the wood.

To overcome the shortcomings of the traditional medium-density fibreboard, a novel ultrasonic-assisted wheat straw pulp fibreboard (UPB) was produced using UWP, and a fire-resistant waterproof coating was obtained *via* a sol-gel process: methyl-POSS and APP were combined as fire retardants and added to the reaction of TEOS and FAS. Strength and water-proof properties, as well as fire resistance were measured.

EXPERIMENTAL

Materials

Wheat straw was obtained from the Shandong province (Liaocheng) of China. The straw was cut into small pieces (with lengths of 2 cm to 3 cm), and then processed with an ultrasonic-assisted pulping treatment in an ultrasonic reactor (self-made, 5-L volume). The UWP pulping methods were in line with relevant literature (Xing *et al.* 2017). Ethanol, tetraethylorthosilicate (TEOS), and ammonium hydroxide (28% in water) were obtained from the Sinopharm Company (Shanghai, China). Tridecafluorooctyltriethoxysilane (FAS) was supplied by Nanjing Quanxi chemical company (Nanjing, China). The polyhedralmethyl-silsesquioxane (methyl-POSS) and ammonium polyphosphate (APP) were purchased from Guangzhou Batai chemical company (Guangzhou, China).

Methods

The preparation method of ultrasonic-assisted wheat straw pulp fibreboard (UPB) was the following: a concentration of 10% to 20% UWP was uniformly mixed and hot pressed using a four-column hot pressing machine (160T, with screen, Zhengzhou Jinxi Machinery Manufacturing Co., Ltd., Zhengzhou, China). The optimum parameters of hot press moulding were a temperature of 80 °C, pressure of 10 MPa (average 5024 N/m^2), and 2-cm thickness.

The strength properties were measured *via* an electronic universal testing machine (CTM8050, Shanghai Association for Instrument Technology Co., Ltd., Shanghai, China).

The preparation steps of the flame-resistant superhydrophobic sol solution were: firstly, two silane precursors, 5 mL of TEOS and an appropriate amount (0.5 mL to 2 mL) of FAS were dissolved in 25 mL of ethanol. The solution was dropped into a mixture solution (6 mL of 28% $NH_3 \cdot H_2O$ in 25 mL of ethanol) and stirred intensively at room temperature for 10 h to conduct a polymerization reaction. Then, the fire retardants (methyl-POSS and APP) were added into this silica sol solution to form a fire-retarding silica sol solution. 0.1 g to 0.5 g of polyhedral methyl-POSS and 0.3 g to 1.2g of APP were added and stirred continually for 1 h to produce the sol solution.

To allow for a comparison, the obtained superhydrophobic sol solution without and with flame resistance were ultrasonically dispersed (KQ-600DE, Kunshan ultrasonic instrument Co., Ltd., Kunshan, China) for 30 min to achieve a homogeneous suspension, and then coated on the surface of a cleaned UPB *via* brushing. Lastly, the treated substrates were dried at room temperature and further cured at 110 °C for 1 h.

The coating procedures scheme of the flame-resistant superhydrophobic surface is illustrated in Fig. 1.

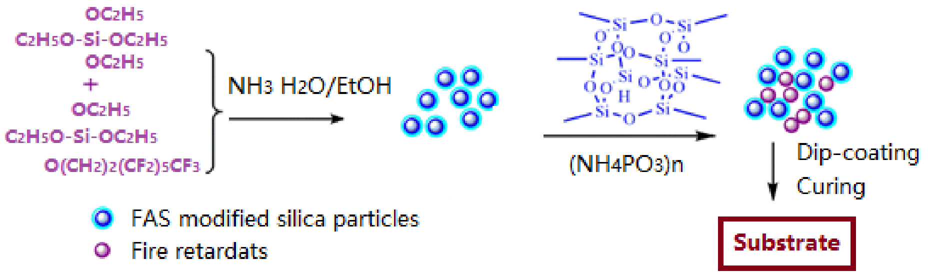

Fig. 1. Schematic of the preparation of the flame-resistant superhydrophobic sol solution and the coating procedures

The SEM images were obtained using a scanning electron microscope (Nova Nano SEM450, FEI, Hillsboro, OR, USA).

The water contact angles (WCA) were measured with a static drop contact angle and interface tension measurement instrument (JC2000A, Shanghai Fangrui Technique Equipment Co., Ltd., Shanghai, China).

The limiting oxygen index (LOI) was measured with a limiting oxygen index measurement instrument (Chengde Kecheng Testing Instrument Co., Ltd., Chengde, China).

RESULTS AND DISCUSSION

Strength Properties of UPB

The UWP was moulded to the fibreboard without adhesives, while the hydrogen bonds could replace the adhesive bonding force to achieve good strength. The strength properties are shown in Table 1, in which the static bending strength, elastic modulus, and internal bond strength of UPB all clearly exceeded the industry standard of general purpose medium-density fibreboard in regular condition (MDF-GP REG), according to GB/T 11718 (2009). These results indicated that as a substitute for wood chips, UWP has a bright prospect to make general-purpose medium-density fibreboard.

Table 1. Strength Performance of UPB comparing to MDF-GP Standard in Regular Condition

Properties	Thickness (mm)	Static Bending Strength (MPa)	Elastic Modulus (MPa)	Internal Bond Strength (MPa)
UPB	2	36.8	3341	0.78
Standard	2	27	2700	0.60

SEM Measurement

To investigate the variation of the surface morphology before and after coating the fire-retarding silica sol solution, an SEM measurement was conducted. Figures 2a, 2b, and 2c show images of the UPB uncoated, coated with the superhydrophobic sol solution without the fire retardant, and coated with the superhydrophobic sol solution with the fire retardant, respectively. The uncoated fibreboard displayed a relatively smooth, continuous structure. After being coated, the surface of the UPB became coarse with a hierarchical structure morphology, which provided nano-scale roughness. Moreover, compared with the superhydrophobic sol solution without the fire retardant, the sol solution with the fire retardant presented a more complex hierarchical structure because the fire retardant added complexity to the surface structure. Furthermore, as presented in Fig. 2d, the average particle size of the FAS modified silica particles was approximately 100 nm to 200 nm. Liu *et al.* (2015) and Ali *et al.* (2016) obtained similar conclusions that the coating surface exhibited a rough, wrinkled, hill-like morphology similar to the microstructure of the lotus leaf (rough micro-scale papillae), which is promising for superhydrophobicity.

To elucidate the origination of the surface morphology of the fibreboard coated with the superhydrophobic sol solution without the fire retardant, the appearances of the samples fabricated by hydrolyzing the two silane precursors in different ratios were explored. When the TEOS content was less than five times that of the FAS, a comparatively clear resin was observed (Fig. 2e). As the TEOS content increased, the liquid became increasingly muddier (Figs. 2f and 2g). The distinction of the appearances with different FAS concentrations was explained by the different hydrolysis rate between the TEOS and FAS. During the solvent's evaporation, the porous framework grew in a succession fashion to form a spanning-cluster, which connected the space of the system. Certainly, the structure of the framework might have been tuned by the reaction conditions. The TEOS quickly hydrolysed into silica particles that turned the solution milky, serving as backbones of the FAS resin framework. As a result, the superhydrophobic property was promoted.

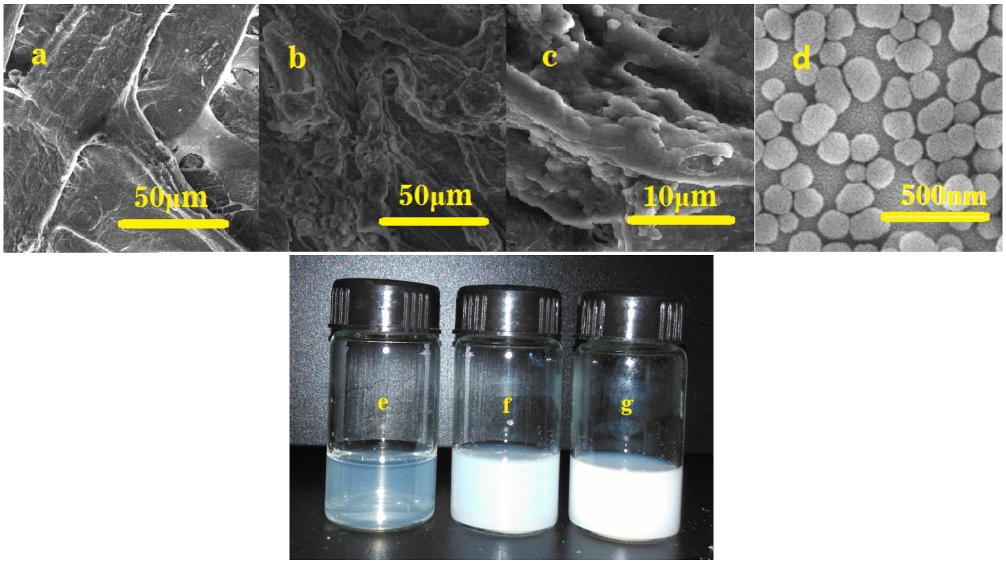

Fig. 2. SEM images of the uncoated fibreboard (a), the fibreboard coated with superhydrophobic sol solution without flame-retardants (b),the fibreboard coated with superhydrophobic sol solution with flame-retardants (c), and the image of the FAS modified silica particles (d); the photograph of EtOH solution of TEOS/FAS sol at different ratios of TEOS/FAS: (e) 5:1, (f) 10:1, and (g) 30:1

Hydrophobic Properties

Water contact angle (WCA) measurement

To investigate the superhydrophobility, the WCA measurement was performed for the uncoated fibreboard (Fig. 3a), the fibreboard coated with superhydrophobic sol solution without fire retardants (Fig. 3b), and the fibreboard coated with superhydrophobic sol solution with fire retardants (Fig. 3c). As shown in Fig. 3a, when dropped on the pristine fibreboard, the water completely permeated into the fabric with a WCA of 61°. Figures 3b and 3c revealed that both the WCAs of the coated surface with and without fire retardants were larger than 150°, which indicated that the fire retardants had little effect on the superhydrophobicity.

The superhydrophobic silica sol solution was prepared by the co-hydrolysis and condensation polymerization of two silane precursors, TEOS and the fluorine-containing silane FAS. During the preparation processes, the effect of the fire retardants on the superhydrophobic property was also considered. The methyl-POSS showed hydrophobic properties due to the methyl groups. The APP is a hydrophilic compound with chain-end groups that have affinity towards water. Therefore, the APP with a higher degree of polymerization ($n > 30$) was applied in the process to decrease the hydrophilic property by reducing the ratios of the chain-end groups. Conventionally, superhydrophobic surfaces are achieved by means of low surface energy in conjunction with multi-scale roughness (Shen *et al.* 1969). According to the SEM images, the superhydrophobicity was derived from a hierarchically structure, which increased the roughness of the surface. Generally, a liquid droplet contacting on a rough surface can be described by the Cassie-Baxter state model. The Cassie-Baxter state model was utilized to elucidate the recombination contact between the liquid droplet and the surface. The liquid droplet could not fill the groove of the surface, allowing air to appear between the liquid droplet and the surface. Consequently, the rolling resistance had been decreased considerably, leading to a lower sliding angle and higher apparent contact angle. Ma and Hill (2006) and Marmur (2003, 2004, 2008) have also explained the significance of the hierarchical structure of surfaces that can support low surface tension liquids from a theoretical understanding. Additionally, superhydrophobic thin films with other properties, such as optical transparency, mechanical flexibility, and fast regeneration visible light transmission (Akira *et al.* 2000; Chen *et al.* 2015) could also be prepared through sol-gel process of the reaction of silane precursors.

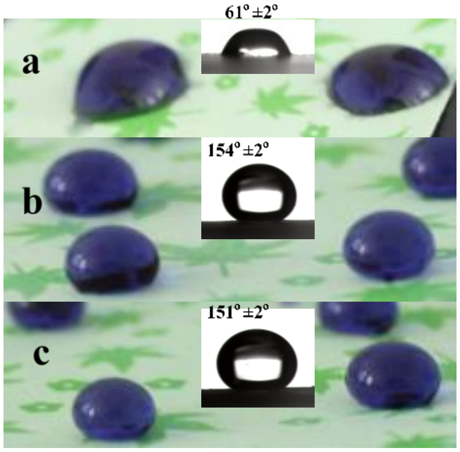

Fig. 3. The WCA of the uncoated fibreboard (a), the fibreboard coated with superhydrophobic sol solution without fire retardants (b), and the fibreboard coated with superhydrophobic sol solution with retardants (c)

Resistance to water penetration

Essentially, a superhydrophobic surface is not only supposed to increase its surface contact angle, but also it is expected to improve resistance to water penetration. The anti-permeability will be influenced by the surface energy of the compound on the surface, and the capillary force that originates from the surface. To explore the resistance to water penetration, the time-resolved WCA measurement was performed. As shown in Fig. 4, the time-resolved WCA curves corresponded to the fibreboard coated with the superhydrophobic sol solution without and with fire retardants. The gentle declining trend of the curves revealed that the water penetration's velocity was slow, which suggested good water anti-penetration properties. It was worth noting that the time-resolved WCA curve of the superhydrophobic sol solution that contained the fire retardants displayed a flatter curve compared to the one without fire retardants, which revealed that the fire retardants had improved the resistance to water penetration. The fire retardants had a positive impact on the surface roughness because they served as the support-bodies of the FAS resin framework. Meanwhile, the abrasion resistance property of super-hydrophobic films could also be effectively improved from the combination of different roughness levels (Tomoki *et al.* 2009).

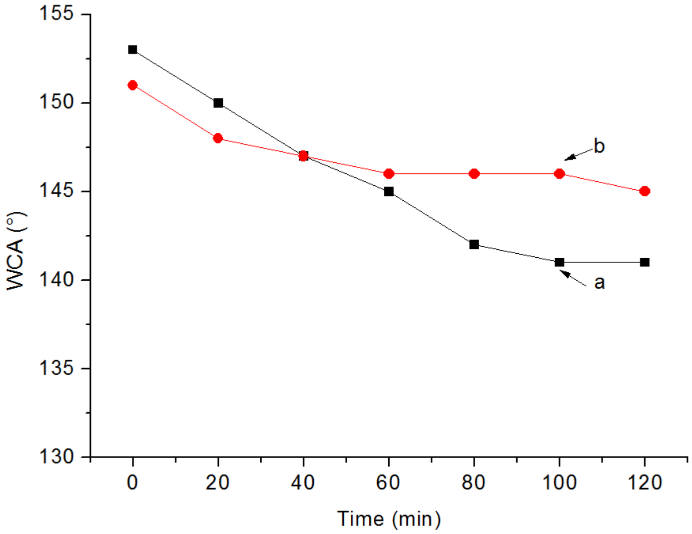

Fig. 4. The time-resolved WCA curves with respect to fibreboard coated with superhydrophobic sol solution without fire retardants (a), and fibreboard coated with superhydrophobic sol solution with fire retardants (b)

Flame Retardancy

The limiting oxygen index (LOI) is one of the most important parameters for describing the flame retardancy of materials with relatively simple determination. The higher the LOI number, the better the flame retardancy quality. Based on the standard GB/T 2406.2 (2009), materials with LOI values below 21% are classified as "combustible," whereas those above 21% are classified as "self-extinguishing." Figure 5 demonstrates that the LOI of the flame-resistant superhydrophobic sol solution was around 23.5% to 25%, which was promoted 5% to 7% compared to the original sample with the LOI of 18%.

To investigate the effect of different fire retardants concentrations on the flame-resistant properties, the LOI of coated paper with varied APP concentrations (same methyl-POSS concentration) (Fig. 5a) and varied methyl-POSS concentrations (same APP concentration) (Fig. 5b) were measured (FAS:TEOS was 1:10). As the high-efficient fire retardants, during the flaming process, APP and POSS could not only form a thin membrane that wrapped tightly around the materials to isolate oxygen, but it also performed a dehydration and carbonization reaction with the combustion materials to decrease the temperature and reduced the release of flammable gases (Song *et al.* 2012; Zhang *et al.* 2017). Typically, when melt-blended with polymers, APP acted as a nucleating agent, decreasing tensile strength (Douglas *et al.* 2014). Therefore, we kept it in the surface film *via* the coating method.

It was illustrated that the LOI was more strongly dependent on the APP concentration than methyl-POSS, because the APP made a predominating contribution to the anti-flaming. The LOI showed a rising trend with the increase of APP concentration (Fig. 5a). However, as the APP concentration reached a certain amount, the LOI was almost constant, due to the limitation amount of the fire retardants attached to the surface of samples.

Figure 5b indicates the effect of the methyl-POSS concentration on LOI. The LOI varied and was not conspicuous with the increase in methyl-POSS concentration, which attributed to the subordinate role to the flame retardancy, compared to APP. However, the retarded combustion phenomenon became clearer with the increase in the methyl-POSS concentration. The methyl-POSS with 0.5%, 1.0%, 1.5%, and 2.0% concentration delayed the flaming time 3 min, 3.5 min, 3.8 min, and 4 min, respectively, which was also a substantial effect.

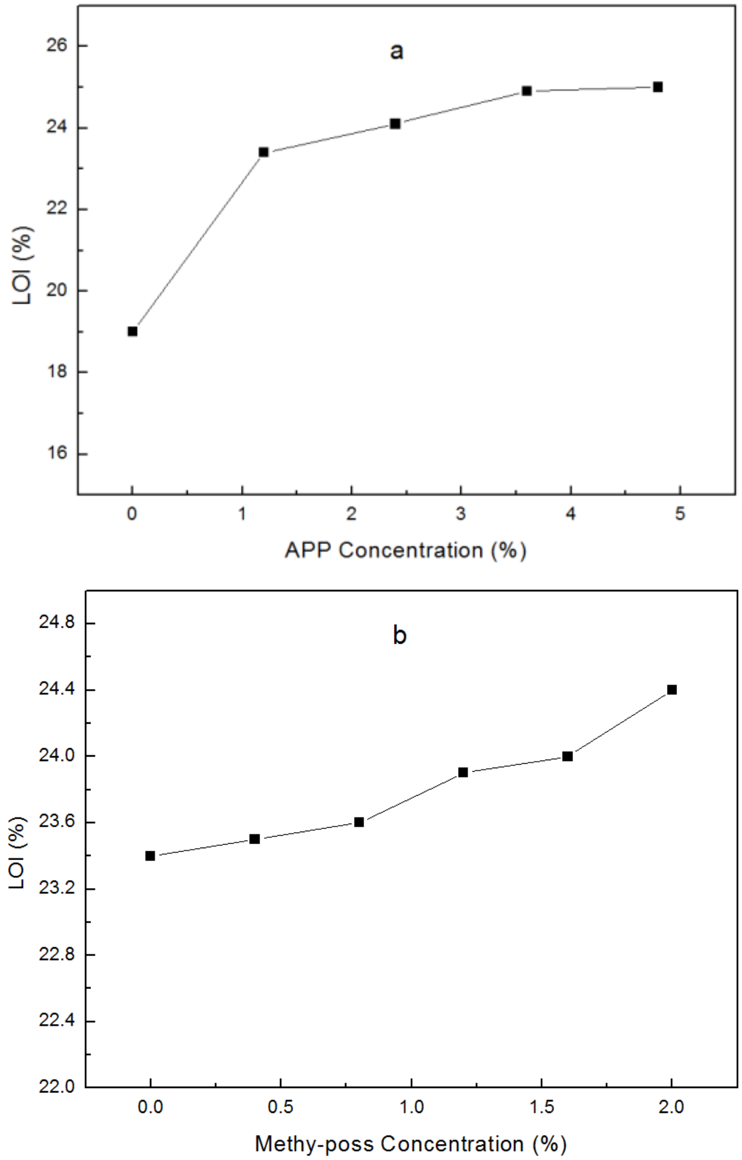

Fig. 5. The limiting oxygen index (LOI) of fibreboard coated with superhydrophobic sol solution with fire retardants with varied APP concentrations (same of methyl-POSS concentration) (a) and varied methyl-POSS concentration but same of APP concentration (b)

CONCLUSIONS

1. A novel medium-density fibreboard produced by ultrasonic-assisted wheat straw pulp (UWP) was fabricated by a simple direct process. It exhibited good strength properties without adhesives, as well as excellent hydrophobicity and flame-resistant properties.
2. The static bending strength, elastic modulus, and internal bond strength of coated UWP medium-density fibreboard (UPB) all clearly exceeded the industry standard for general purpose medium-density fibreboard, while it showed promise a new prospect of UWP as a substitute for wood chips to produce medium-density fibreboard.
3. The SEM and the contact angle measurements revealed that the superhydrophobicity originated from the hierarchical structure morphology. The sol solution coating procedure had given the original hydrophilic fibreboard superhydrophobic properties. The time-resolved water contact angle measurement suggested excellent resistance of water penetration, and the flame retardants had improved the resistance of water penetration to some degree.
4. The limiting oxygen index of fibreboard samples coated with the sol solution increased from 18% to 24%, which indicated that the sol solution provided the materials with outstanding flame retardancy.

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