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ARTICLE TYPE

Preparation of gypsum/sawdust green composite with spray coating

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This paper addresses the development of green composite from gypsum and sawdust (SW) by using spray coating technique with water-based epoxy (WBE). Positive effect of SW water extractives on gypsum was found by the addition of antifoam agent. Mechanical testing shows that the SW water extractives could increase the flexural and compressive strength of gypsum by 10% and 7% respectively. XRD characterization shows the crystallinity of gypsum has been improved by adding SW water extractives. Mechanical testing of gypsum/SW composite shows that the lightweight composite with promising mechanical performance could be obtained by the WBE spray coating; the flexural and compressive strength of composite are 4.6MPa and 13.3MPa respectively for 20% SW addition, and 3.4MPa and 8.7MPa respectively for 30% SW addition.. Attenuated total reflectance-fourier transform infrared (ATR-FTIR) characterization shows that 1) WEB could be coated on the surface of SW particles by using spray coating; 2) the coated WEB would reduce the water uptake of SW. The further analysis with optical microscopy (OM)-Image Pro Plus demonstrates that the reduction of water uptake has led to an increase of the gypsum covering ratio from 42% to 68%. Field emission gun-scanning electron microscopy (FEG-SEM) characterization again illustrates that WBE coating has improved the interfacial properties of SW with gypsum and kept high gypsum adhesion even in high relative humidity (RH) condition.

1 Introduction¹

Lignocellulosic biomass offers benefits to the environment due to its renewability and carbon-neutrality¹ and is becoming an attractive resource for biomass industry. As one of them, sawdust (SW) is the by-products from the mechanical milling or processing of timber (wood) into various useable sizes. Up to 24.15 million cubic meters per year of this waste are produced. Lamentably, most of them are either burnt or land filled, bringing about various environmental problems like air pollution, emission of greenhouse gases and occupation of useful land. The disposal of SW is getting more and more attentions in recent years. While this forest waste is usually developed as adsorbents²⁻⁵ and fuels⁶⁻⁸, a variety of practical applications have also been attempted, such as, in making inorganic-bonded composites⁹⁻¹¹, filler of polymers¹²⁻¹⁷, composting or fertilizer¹⁸⁻²⁰ and chemical intermediates^{1, 21-23}. Among

these disposals, inorganic-bonded SW, as well as other lignocellulosic particles²⁹⁻³², composites which combine SW with such mineral as Portland cement^{24, 25}, magnesite cement²⁶ and gypsum^{10, 27, 28} have been a long history of development. The first commercial inorganic-bonded (cement) SW composite which was used as building materials dating back to 1930s³³. They have been used primarily as interior wall and ceiling panels due to the light weight, good sound and thermal insulation properties. By the early 1960s, a high-density cement-bonded particleboard was developed leading to wide applications³⁴ of inorganic-bonded SW composite. Nowadays, these composites are used as exterior walls³⁵⁻³⁷, roof shingles³⁸ and tiles³⁹ for building applications.

Gypsum-bonded SW composite was developed relatively late, which started from 1980s¹⁰. Two main forms of gypsum, namely, α -⁴⁰ and β -gypsum^{27, 41} are always used as the matrix. The β -gypsum is the popular one due to its easy manufacturing process and low energy cost. The gypsum-bonded SW composites get less attention for a long time due to their low mechanical performance, low durability and bad compatibility⁴²⁻⁴⁴ with SW. Fortunately the green characteristics of gypsum, e.g. lower embodied energy⁴⁵ and lower embodied carbon⁴⁶ are realized in recent years due to the growing awareness of the interconnectivity of global environmental factors⁴⁷, and

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the gypsum-bonded SW composites have been revisited. A promising report from Henke⁴⁸ showed that SW and gypsum would have potential application in 3D building print.

The issue of low strength is still challenging the application of inorganic-bonded SW composites. Efforts have been done with the attempt to overcome this issue by 1) mixing with synthetic fibres^{28, 49}, other natural fibres^{40, 50-52} and nanomaterials, such as SiO₂⁴², cellulose nanofiber⁴⁴; 2) the modification of matrix and 3) modification of SW. The 2) and 3) are the essential part of these efforts. The main modifications of the matrix in the previous reports, including 1) an addition of water soluble polymers⁴⁰ to increase the mechanical performance of the matrix; and 2) addition of calcium chloride^{24, 53, 54} to enhance compatibility between SW and matrix. While these chemical modifications could bring about the improvement of mechanical performance of composite, they will consume amount of additives (more than 5 % of matrix by weight). As for the modification of SW, it was found that the extractives from SW would have adverse effects on the exothermic hydration (or setting) of cement²⁴ or gypsum²⁷. The reported modifications include 1) the removal of SW extractives, e.g. hot water treatment^{24, 55}, alkaline treatment^{9, 24, 41, 55}; and 2) reduction of the release of wood extractive by coating with waterproofing materials, e.g. cutback asphalt⁵⁶ and varnish⁵⁶. The first method dominates the modification of SW up to 30 years; it can remove the hot water soluble extractive, but it consumes energy to heat the water and requires water to wash the SW with the pitiful increase in mechanical performance of composite. In addition, this method is not in favor of the making of high strength inorganic-bonded SW composites; the second method could obtain the SW composites with high strength, but it consumes waterproofing materials, which are up to 25% of SW by weight. Finding a solution to overcome these issues would open up the possibility of revival of inorganic-bonded SW composites.

In this present paper, we report a novel modification of SW by using spray coating of WBE on SW to 1) overcome these issues as mentioned above and 2) make high mechanical performance of lightweight gypsum/SW composite: SW water extractives were firstly characterized by using ATR-FTIR; the effect of SW water extractives on the performance of gypsum was then investigated; the efficacy of spray coating on the mechanical performance of composite was further studied; and finally, the mechanism of the reinforcement with spray coating was revealed by the interfacial investigation with OM and SEM characterizations.

2 Materials and Methods

2.1 Materials

SW was donated by Wood Workshop of Brunel University. ADVA650 (superplasticizer) was kindly donated from Grace Construction Products Ltd, UK. Hemihydrate gypsum was supplied by Bentley Advanced Materials Ltd, UK. Potassium citrate tribasic monohydrate (KCTM) and silicone antifoam were supplied by Sigma-Aldrich. WBE was order from New Venture Products Ltd, UK.

2.2 Extract of SW extractives

Cold water extractives of SW were carried out as following procedure: SW particles (0.5-1.0mm) were immersed in distilled water for 24h at room temperature with 0.275 SW/water (s/w) ratio; the solutions were the filtered off by using a Buchner funnel, filter paper and a heavy-wall filtering flask (2 L) connected to vacuum pump. 1000 ml of filtrates (fs) was collected for the work discussed in the section 2.4.

2.3 Spray coating of SW

The WBE is a two part (part A and part B) product. 3g of part A was mixed with 12g part B; the mixed WBE was then diluted by 100 ml water and stirred for 10 min prior to coating. 600g of raw SW (size 0.5-1.0mm) was then coated with the diluted WBE by sprayer in the mixer. The coated SW was then dried with vacuum oven at 70°C for 5h and conditioned at 20±2°C and 0% RH before using.

2.4 Preparation of gypsum board and gypsum/SW composite

In order to investigate the effect of SW on the mechanical performance of gypsum, two kinds of gypsum board were prepared: 1) gypsum/SW water extractives and 2) gypsum/SW water extractives/antifoam agent. The former was prepared as the following procedure: 360g fs (360g) which was obtained from section 2.2 were used to mix with 850g gypsum and 190g distill water; the gypsum mortar was then stirred for 2 minutes; the gypsum mortar was then cast in the mould (40x40x160mm) under room temperature (about 22°C); after casting, all the samples were kept in the laboratory at room temperature for 28 days. The latter was prepared with a similar procedure; a slight difference was the addition of antifoam agent (0.1% by the weight of gypsum) during the stirring process. The reference boards of 1) gypsum and 2) gypsum/antifoam agent were prepared (gypsum/water ratio 1.54).

Gypsum/WBE coated composite was prepared with various SW addition, namely, 20% and 30% (by the weight of gypsum). Before testing these composites were prepared according to EN 13279-2:2004, the other parameters for preparing these composites were gypsum/water ratio 1.67; 0.9% addition of ADVA 650.

The reference boards of 1) gypsum/ADVA 650 (the gypsum/water ratio is 2.5) and 2) gypsum/raw SW/ADVA 650 were also prepared. Densities of gypsum, various gypsum/SW composites were measured according to ASTM C0567.

2.5 Mechanical testing

The flexural and compressive strength of samples which were obtained in section 2.4 were determined using an Instron 5566 with 150 KN load capacity. Flexural strength measurement was tested by Instron equipped with a flexure device which conforms to EN 196-1, a load was applied vertically by means of a loading roller to the opposite side face of the prism with a rate of 10 N/s until the fracture. The compressive strength was measured by Instron equipped with compression device to test portions of 40x40x160 prisms broken in flexure to EN 196-1. A load was applied and increased smoothly at the rate of 800 N/s over the entire load application until the fracture. The flexural and compressive strength are calculated according to the expressions given in EN 13279-2:2004. For flexural strength, three samples were calculated and six samples were calculated for the compressive strength.

2.6 XRD characterization

Gypsum, gypsum with addition of antifoam agent and mixture of gypsum-antifoam agent-SW extractives were subjected to a powder X-ray diffraction method analysis (PXRD) respectively. For this analysis, a D8 advanced Bruker AXS diffractometer, Cu point focus source, graphite monochromator and 2D-area detector GADDS system were used. The diffracted intensity of CuK α radiation (wavelength of 0.1542 nm) was recorded between 5° and 60° (2 θ angle range) at 40 kV and 40 mA. Samples were analyzed in transmission mode.

2.7 ATR-FTIR characterization

Fine SW particles with size 0.2-0.3mm were coated as described in the Section 2.3. The coated particles were then dried in oven at 103 \pm 2°C for 3h. Then the reference samples (raw SW with size 0.2-0.3mm) and modified samples were mounted on an ATR equipped with 3 \times bounce diamond crystal and an incident angle of 45° was used. ATR-FTIR spectra were recorded on a PerkinElmer Spectrum one Spectrometer. The instrument was operated under the following conditions: 4000-650cm⁻¹ range; 4cm⁻¹ resolution; 16 scans and 20°C. Deconvolution of spectra was carried out as previous report⁵⁷.

2.8 Microscopy Characterization

OM and FEG-SEM were used in this present work to characterize the interfacial properties of SW. By using OM, the gypsum covering ratio on the surface of sawdust was investigated. The loose sawdust particles

from the fracture sections were collected carefully (20 particles was collected from each fracture section). The particles were then characterized by BX51 Reflected Light Microscope with 5 \times and saved as uncompressed color images (JPEG format). The area of gypsum covering (A_{gy}) and the area of sawdust (A_{sw}) were counted with Image-Pro Plus 6 (Media Cybernetics Inc., Bethesda, MD, USA) according to the different color between gypsum and sawdust. Then the gypsum covering ratio was worked out by the following equation:

$$C_{gy} = \frac{A_{gy}}{A_{sw}} \quad (1)$$

Where, C_{gy} is the gypsum covering ratio, A_{gy} is the area of gypsum and A_{sw} is the area of sawdust.

Specimens from 1) gypsum/raw SW/ADVA 650 and 2) gypsum/WEB coated SW/ADVA 650 were cut into thin sheet by using a cutter (Delta PetroCut, Buehler, UK). The cut sheet were then polished 1200-grit sandpaper, using grinder-polisher (ECOMET 6, Buehler, UK) at a speed of 100rpm with a force of 50N; the polished specimens were then cleaned with ultrasonic bath for 5 minutes. After drying in room temperature, the clean composite sheets were then examined with a Zeiss Supra 35 VP field emission scanning electron microscopy (FEG-SEM). The test pieces were coated with thin layer platinum on the surface in an Edwards S150B sputter coater to provide electrical conductivity. Following coating, samples were observed and operated at 10 kV using the secondary electron mode with images collected digitally.

3 Results and Discussion

3.1 SW water extractives

Extractives from wood particles are a broad class of low-molecular weight organic compounds that are soluble in polar or non-polar solvents. In the process of making inorganic-bonded SW composites, the main extractives are water soluble compounds (e.g. tannin, acetic acid)⁵⁸. It has been reported that wood extractives could 1) retard the hydration of the inorganic binders^{27, 58} and 2) alter crystalline structures of binders⁵⁹. It has been reported that the SW water extractives can reduce the mechanical performance of cement-bonded SW composite significantly⁶⁰ due to the present of glucose⁶¹ and slight reduction on gypsum-bonded SW composite²⁷. In this present work, we investigated the effect of SW water extractives on the mechanical performance of gypsum by adding antifoam agent. The results are shown in Table 1. It can be found that: 1) without an addition of antifoam agent, the addition of SW water extractives would decrease the mechanical performance of gypsum; 2) without addition of SW

water extractives, the addition of antifoam agent would decrease the mechanical performance of gypsum slightly; 3) surprisingly, with the addition both of antifoam agent and SW water extractives, the mechanical performance of gypsum could be increased; the flexural strength and compressive strength are increased by 10.38% and 6.99% respectively. In cement system, polysaccharides (e.g. starch, hemicellulose) would be partially converted to OH-C-C=O group due to the high alkali condition⁶²; this active adsorbing group has great stability in alkaline solution and displays weak set-retarding property, and it is the main factor that reduces the mechanical performance of cement⁶³. However, this oxidation would not possibly appear in gypsum system due to the weak acidity of gypsum paste (pH ~6.5). Therefore, it can be concluded based on these results that: 1) SW water extractives may contain foaming agent which may be attributed to tannin; tannin has surface active properties⁶⁴; 2) this foaming agent may be the main factor that reduces the mechanical performance of gypsum; 3) by eliminating the foaming effect of foaming agent with antifoam agent, the SW water extractives could increase the mechanical performance of gypsum.

Table 1 Effect of SW water extractives on the mechanical performance of gypsum

Materials	Flexural strength (MPa)	C.V. (%)	Compressive strength (MPa)	C.V. (%)	Density (g/mm ³)
Gypsum	4.43	7.91	10.59	2.02	1.13
Gypsum + fs	3.74	8.71	9.85	2.37	1.12
Gypsum + 0.1% antifoam agent	4.37	9.14	10.53	3.21	1.14
Gypsum + fs + 0.1% antifoam agent	4.89	4.19	11.33	3.13	1.16

Gypsum pastes with and without the addition of SW water extractives after antifoam treatment are examined by XRD. As shown in Fig. 1, the gypsum peaks for 1) raw gypsum, 2) gypsum with addition of antifoam agent and 3) gypsum with addition of antifoam agent and SW water extractives are all well defined. It is apparent from Fig. 2 that: 1) an addition of antifoam agent would result in a decrease of intensity which means the formation of

weaker crystallinity inside gypsum and 2) interestingly, the addition with both of antifoam agent and SW water extractives give rise to an increase of intensity which means the formation of higher crystallinity inside gypsum. These results are in agreement with the mechanical testing as described previous.

Fig. 1 X-ray diffractogram of gypsum with and without SW water extractives addition after antifoam treatment

3.2 Modification of SW

Generally, epoxy is mixed with cement mortar and has been widely used as a type of polymer cement mortar due to its high bond strength to cement concrete, high inherent strength, water resistance, chemical resistance and weather-ability⁶⁵. In addition, the epoxy has also been used to improve the water resistance of gypsum by mixing with gypsum⁶⁶ or coating on the surface of gypsum⁶⁷. In present work, WEP was used to modify SW to reduce the water uptake of SW.

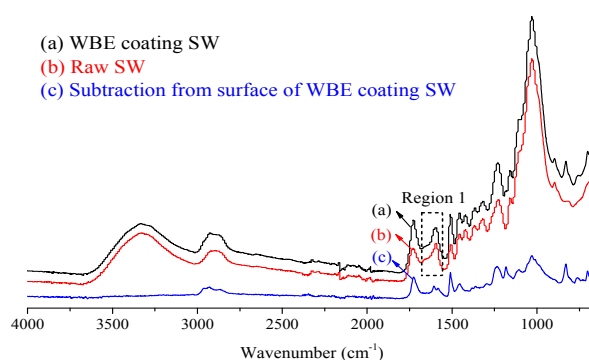


Fig. 2 ATR-FTIR spectra of WBE coating SW, raw SW and subtraction from surface of WBE coating SW

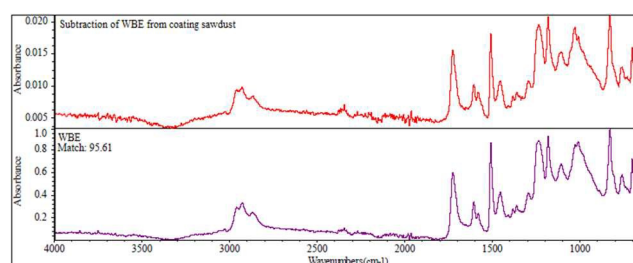
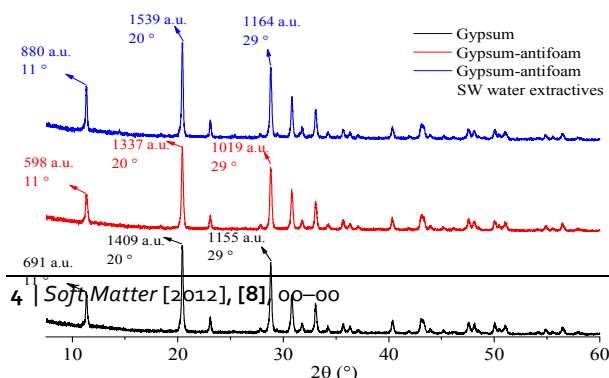


Fig. 3 Matching result of subtraction from surface of WBE coating sawdust with OMNIC software

The ATR-FTIR spectra of (a) WBE coating SW, (b) raw SW and (c) subtraction from surface of WBE coating SW were presented in Fig. 2. The obtained spectrum (c) was then compared with the WEB spectrum which has been gathered and added into the OMNIC library by search program. The search result as shown in Fig. 3 illustrates that the spectrum (c) has an excellent match (match value 95.61) with WBE. This indicates that WBE could be coated on the surface of



SW by using spray coating.

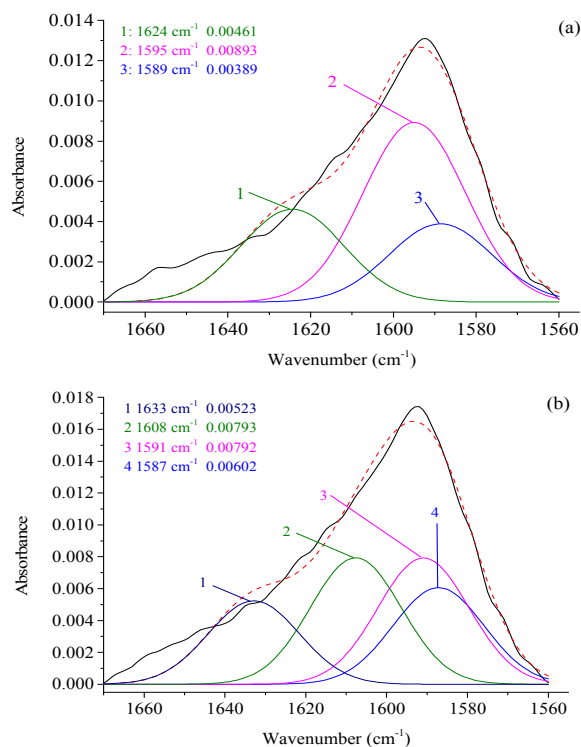


Fig. 4 Deconvoluted ATR-FTIR spectra from 1670 to 1560 cm⁻¹ for SW without treatment (a) and WBE spray coating (b)

In addition, the absorbed WEB on SW by using spray coating can also be evidenced from the deconvolution of Region 1 in Fig. 2. The deconvolution results are shown in Figs. 4 (a) and 4(b). As shown in Fig. 4(a), C=O stretching of lignin⁶⁸ in 1588 cm⁻¹ and 1624 cm⁻¹ and OH bending of absorbed water⁶⁹ in 1595 cm⁻¹ can be observed. After spray coating treatment, a new band in 1608 cm⁻¹ which may be attributed to the stretching of epoxy ring⁷⁰ was observed (see Fig. 4(b)). Meanwhile, a significant decrease of absorbance from water band can also be found from Fig. 4(b). After spray coating, the absorbance of water band decreases from 0.00893 to 0.00792. This indicates that the WBE treatment would decrease the water uptake of SW particles. It is recognized in the literature that the mechanical performance of composite can be significantly influenced by surface properties of filler and interface between filler and matrix. For lignocellulosic materials based composite, water content on the filler surface and interfacial properties are the main factors which influence the mechanical performance of composite. It had been known that water could infiltrate between the crystals of gypsum and partially shield the bonds, this would weaken the solid structure of gypsum⁷¹; therefore, the reduced water uptake would 1) increase the mechanical performance of gypsum around SW and 2) increase the interfacial properties between SW and

gypsum.

Table 2 Effect of SW modification on the mechanical performance of gypsum/SW composite (with ultrasonic pretreatment)

Materials	Flexural strength (MPa)	C.V. (%)	Compressive strength (MPa)	C.V. (%)	Density (g/mm ³)
Gypgsum (ADVA 650)	6.43	7.49	29.14	5.72	1.50
Gypgsum + raw SW (20%) + ADVA 650	2.66	2.39	7.01	4.19	1.10
Gypsum + SW (20%, 0.5% WBE coating) + ADVA 650	4.59	2.89	13.25	2.22	1.11
Gypsum + SW (30%, 0.5% WBE coating) + ADVA 650	3.36	8.85	8.73	8.98	0.87

Traditionally, the modification of SW was carried out by using hot water treatment^{24, 55} and alkaline treatment^{9, 24, 41, 55}. High water/SW ratio and high temperature are required for these traditional modification processes; this would consume amount of water and energy. In addition, high water absorption and low mechanical performance of mineral-bonded SW composite have still not been overcome. In this present work, we modified SW with novel method, namely spray coating, which was carried out under low water/SW ratio and room temperature. The influence of WBE coating on the mechanical performance of gypsum/SW composite is shown in Table 2.

As shown in Table 2, 1) an addition of SW would reduce the mechanical performance of gypsum, as described in Section 3.1, the SW water extractives would in favor of the increase of mechanical performance of gypsum, therefore, the reduction of mechanical performance of gypsum with SW may be attributed to the surface properties of SW particles; 2) an improved performance of gypsum/SW composite could be obtained by WBE spray coating: for 20% SW addition, the flexural and compressive strength of gypsum/WBE coating SW composite are 4.59 MPa and 13.25 MPa respectively; compared with gypsum/raw SW composite/ADVA 650, they are increased up to 72.56% and 89.02% respectively, this performance can meet the requirement of building bricks according to ASTM C 62. For 30% SW addition, the composites display light weight (0.87 g/mm³) and promising mechanical performance; the flexural and compressive strength of composite are 3.36 MPa and 8.73 MPa respectively, it should be noted that this performance is higher than the commercial blocks which are widely

used in internal and external leaf of cavity walls, solid walls, separating walls, flanking walls, partitions or/and foundations.

3.3 Effect of spray coating on the interface properties of SW

Examination of the OM micrographs of the SW particles from composite showed two types of color (see Figs. 5(a) and 5(c)): 1) SW itself with taupe color and 2) gypsum cover area with white color. By using Image-Pro Plus software, the covered area of gypsum on the surface could be identified. Fig. 5 presents the processed images for the analysis of the visual characteristics of the SW surface. Areas covered by the contaminants can be outlined or distinguished in a different color, which could be used for area measurements or counting. Fig. 6 shows the percentage area of gypsum on the surface of SW particle.

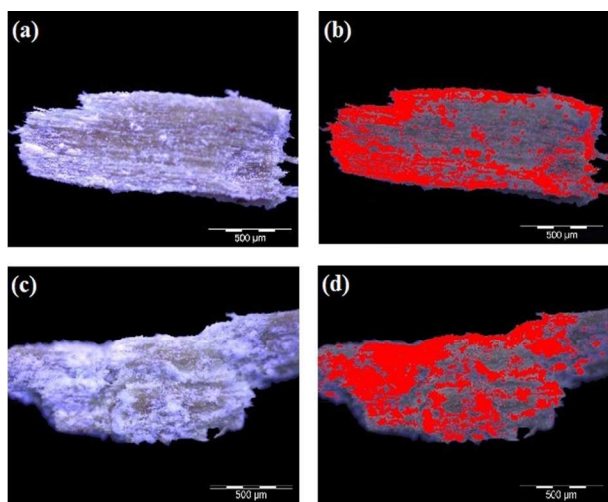


Fig. 5 Identification of gypsum covered area on SW without and with spray coating using Image-Pro Plus image analysis: (a) raw SW, (b) raw SW with Image-Pro Plus image analysis, (c) WBE coating SW and (d) WBE coating SW Image-Pro Plus image analysis

As shown in Fig. 6, after spray coating gypsum cover ratio was increased from 41.79% to 68.43%. This confirms that the reduced water content inside SW after spray coating would increase interfacial properties between SW and gypsum as describe above. FEG-SEM (see Fig. 7) characterization shows that much more and bigger gaps between SW and gypsum could be observed in the composite without WBE spray coating. As describe in Section 2.7, the prepared process of examined composite sheet has used water and ultrasound to remove the powder. This is somewhat like the environment with high RH. The bigger and more gaps inside composite without WBE spray coating also confirm the aforementioned interfacial properties. It is apparent that 1) WBE spray coating would reduce water absorption of SW in high RH condition; 2) higher adhesion of gypsum on SW would be obtained even in

high RH condition and 3) in high RH condition, the composite with WBE spray coating would display higher mechanical performance.

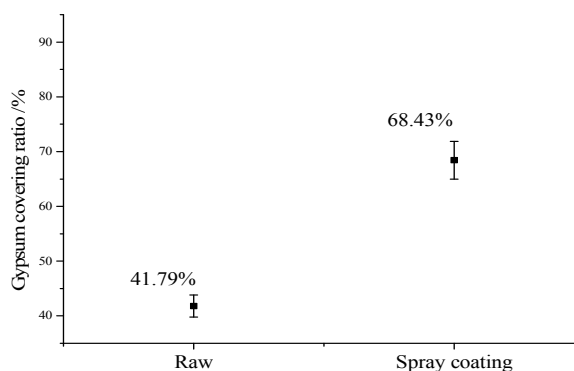


Fig. 6 Gypsum covering ratio on sawdust surface from sawdust/gypsum composites without and with spray coating treatment

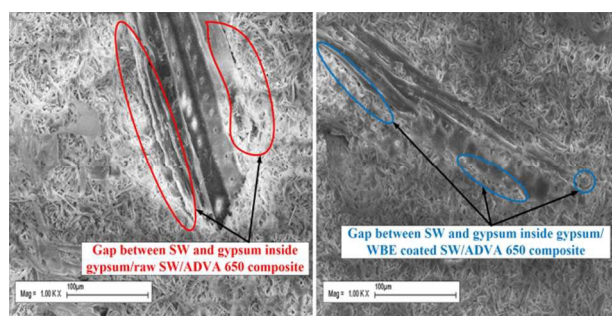


Fig. 7 Gap between SW and gypsum inside (a) gypsum/raw SW/ADVA 650 and (b) gypsum/WBE coated SW/ADVA 650 composites

4 Conclusions

Green composites from gypsum and SW with WBE spray coating are presented. By eliminating the foaming effect of foaming agent, the SW water extractives could increase the crystallinity of gypsum and the mechanical performance of gypsum. SW with less water uptake could be obtained by using WBE spray coating due to the hydrophobicity of WBE which is coated on the surface of SW particles. This would increase the interfacial properties of SW with gypsum and keep high gypsum adhesion even in high RH condition. The most important finding was the improved mechanical performance of gypsum/SW composite could be obtained by using WBE spray coating: for 20% WBE coated SW addition, the flexural and compressive strength of composite are 4.59 MPa and 13.25 MPa respectively; and for 30% WBE coated SW addition, 3.36 MPa and 8.73 MPa respectively.

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