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Thermal property and flame retardancy comparisons based on particle size and size distribution of clays in ethylene vinyl acetate (EVA) adhesive sheets for cross-laminated timber (CLT)

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Abstract

Cross-laminated timber (CLT) is widely used as a building material, and recent interest in this material has increased rapidly due to environmental problems. For instance, the phenol resorcinol formaldehyde (PRF) used as an adhesive in CLT can release toxic gases during a fire, resulting in adverse health effects. These properties cause concern regarding the use of CLT as a building material and restrict its use. In this study, the effects of the particle size and size distribution of clays on the thermal properties and flame retardancy of composites were investigated. Ethylene vinyl acetate (EVA)/clay adhesive sheets were prepared to substitute for the PRF that is used to cohere the timber in CLT. To increase the dispersibility of clay inside the EVA matrix, the clays were organically treated using cetyltrimethylammonium bromide (CTAB), and the interlayer spacing was determined via X-ray diffraction. In addition, the EVA/clay adhesive sheets presented a broad peak due to their good dispersion. As the clay contents increasingly degraded, the thermal properties, such as the degradation temperature and final residue, of the EVA/clay adhesive sheets enhanced. However, the adhesive sheets showed continuous improvement only for a specific particle size and size distribution due to the difference in the degree of organic treatment. The results from cone calorimetry showed a decrease in the heat release rate (HRR) as the clay content increased. With respect to the flame retardancy of EVA/clay adhesive sheets, clay with a large particle size is more effective than clay with a small particle size, while the size distribution does not have a strong influence.

1 Introduction

Cross-laminated timber (CLT) is an engineered wood building material that was introduced in Austria and Germany in the mid-1990s. In the early 2000s, construction using CLT increased significantly and contributed to the green building movement, resulting in increased efficiencies, more product approvals, and improved marketing and distribution channels (Gagnon et al. 2013; Mohammad et al. 2012). In addition,

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given that the interest in natural disasters, such as recent earthquakes and floods, has increased, wooden building materials with structural strengths have been re-evaluated, and the possibility of a new expansion in the building market has been presented. The most widely used interior material for wooden constructions is CLT, which consists of a lumber board, veneer, alternating particle and strand. In previous full-scale fire tests, CLT panels have been shown to have excellent fire resistance comparable to those of typical heavy construction assemblies of reinforced concrete buildings, due to the wood itself and the insulation effect from the formation of the char layer (Gagnon et al. 2013; Emberley et al. 2017). However, the main cause of adverse health effects due to fire is the bronchial damage and suffocation caused by the toxic gas rather than the damage caused by the fire. Phenol resorcinol formaldehyde (PRF) is the most commonly used CLT adhesive, and the formaldehyde in PRF is one of the organic irritants in fires. Recently, research on the use of non-halogen flame retardants was actively conducted to solve the problem of toxic gases generated from halogen flame retardants in the event of a fire. However,

the flame retardant performance of the non-halogen flame retardants does not satisfy the performance of the halogen flame retardants. In addition, Yue et al. (2017) measured the fire resistance of timber with formaldehyde resin using a cone calorimeter and, as a result, confirmed the problem with the generation of toxic gas.

Ethylene vinyl acetate (EVA) is widely used in industrial fields, such as in the wire, cable, wrapper, adhesive and drug industries (Wang et al. 2012a, 2012b). In particular, due to the interest in developing an eco-adhesive with an improved melting index and excellent adhesion, research on an EVA hot melt adhesive (HMA) has been actively advanced (He et al. 2016). However, it is necessary to enhance the fire resistance of EVA due to its low flammability and high calorific value, which can limit its various industrial applications (Hoang et al. 2013; Wang et al. 2012a, 2012b).

Clay is formed by plate-like two-dimensional (2D) nanofillers, and it has been widely used to reinforce various polymers due to its competitive cost, low density and proper stiffness/strength (Ray and Okamoto 2003; Montazeri et al. 2017; Moya et al. 2015). Clay is low in price compared to other synergists, and previous research has regarded it as one of the best flame retardant additives (Tang el al. 2003; Hu et al. 2016). Pristine clay has a high hydrophilicity and is not highly compatible with organic polymers. The low compatibility weakens the dispersibility of the clay, thereby degrading the mechanical, barrier, thermal, and flammability properties of the final product (Jian et al. 2016). For these reasons, studies have been conducted in academia and industry to improve the compatibility between hydrophilic smectite and hydrophobic polymers through the chemical modification of clay surfaces (Kiliaris and Papaspyrides 2010). In this research, pristine clays were organically modified with an alkylammonium cation, which is a typical method to substitute hydrophobicity for hydrophilicity.

In this work, pristine clays with different particle size distributions were organically modified by cetyltrimethylammonium bromide (CTAB), and the samples were analyzed. Following modification, the clays were predispersed in an EVA/toluene solution, and they were subsequently meltmixed with the EVA polymer to prepare an adhesive sheet for CLT. The structure of the CLT via the EVA sheets is presented in Fig. 1. In this paper, the EVA/clay adhesive sheets were only studied to confirm their applicability to CLT. The particle size distribution and zeta potential of the clays were analyzed by a Zetasizer. The organic modification analysis and the clay dispersion in the adhesive sheets were investigated via XRD. Additionally, the thermal properties of the adhesive sheets with different types and contents of clay were determined by thermogravimetric analysis (TGA). Finally, the fire retardancy of the EVA/clay adhesive as a function of clay type and content was assessed using a cone calorimeter.



Fig. 1 CLT structure via an EVA adhesive sheet

2 Materials and methods

2.1 Materials

EVA (VS420) with 21.5 wt% vinyl acetate content, which was supplied by Lotte Chemical Corporation (Republic of Korea), has a density of 0.945 g/cm³, a melting point of 79 °C, and a melt flow index of 2.0 g/10 min. Maleic anhydride-grafted EVA (MA-g-EVA), which was supplied by DuPont, has a density of 0.91 g/cm³, a melting point of 71 °C, and a melt flow index of 1.4 g/10 min. This material was used as a compatibilizing agent to support the clay dispersion in the EVA sheets. In this study, two types of clays and a representative clay that is widely used in industry were selected to compare their properties. The three types of clays used in this research were Laponite XLG (synthetic nanoclay; LAP) and Cloisite Na+ (CSN), from Rockwood Additives Ltd., and S1ME (Na-fluorine) (ME), from Co-Op Chemical Co., Ltd. The clays were dried in an oven at 80 °C for 24 h prior to compounding to remove any residual water in the 'as-supplied' materials. Cetyltrimethylammonium bromide (CTAB, Sigma-Aldrich, USA) was used as an organic surfactant. Toluene was used as a solvent. All of the materials were used as received without further purification.

2.2 Modification and compounding of specimens

2.2.1 Organic modification

The clays were modified as follows. First, 20 g of pristine clay was soaked in 1000 mL of deionized water for 24 h, and CTAB was added slowly. The concentration of added CTAB was 1.0 of the cation-exchange capacity (CEC) of the host clay. The reaction mixture was equipped with a stirrer and thermometer and stirred for 24 h at 80 °C. The final reaction products were separated by centrifugation, freeze-dried, ground and then oven dried for 24 h at 100 °C. The organically modified clay was stored in wide-mouth bottles to avoid moisture infiltration. Figure 2 illustrates the ideal scheme for the clay organic modification from previous research (Yan et al. 2018).



Fig. 2 Outline of the organic clay modification scheme.

2.2.2 Preparation of EVA/clay adhesive sheet

First, the EVA/clay predispersion (ECP) was prepared to prevent the loss of clay and to enhance the powder's dispersion in the EVA/clay adhesive sheets. The EVA solution was prepared by adding EVA to toluene under stirring at 90 °C for 1 h. The clay dispersions were prepared by stirring them in the same solvent for 6 h at 90 °C. The clay dispersion was added to the EVA solution and then continuously stirred for 24 h. The EVA/clay solution was then placed on a silicone release film and heated in an oven at 100 °C for 12 h to evaporate the toluene.

The formulations of the EVA/clay adhesive sheets are listed in Table 1. The EVA/ECP series were blended with

the MA-g-EVA in a laboratory-sized twin-screw extruder (BA19, Bau Technology, Republic of Korea) using three general processes: melt blending, extrusion and pelletizing. The extruder barrel was divided into eight zones, with the temperature in each zone being individually adjustable. The temperature of the mixing zone in the barrel was maintained at 100 °C with a screw speed of 300 rpm. The strand after extruding was cooled in a water bath and pelletized with a pelletizer (Bau Technology, Republic of Korea). The final pellets were oven dried at 80 °C for 24 h and stored in sealed polyethylene bags to avoid moisture permeation. The dried extruded pellets were fabricated to EVA/clay adhesive sheets using a hot press prior to the XRD and cone calorimeter analyses. The temperature of the hot press was maintained

Table 1 Formulations of the ECA/clay adhesive sheets	Sample Names	EVA (wt%)	MA-g-EVA (wt%)	ECP			
				EVA (wt%)	Clays (phr)		
					LAP	CSN	ME
	Neat EVA	100	_	-	_	_	_
	ECA-L01	91	5	4	1	-	_
	ECA-L03			12	3	-	_
	ECA-L05			20	5	-	_
	ECA-L10			40	10	-	_
	ECA-C01	91	5	4	-	1	_
	ECA-C03			12	-	3	_
	ECA-C05			20	-	5	-
	ECA-C10			40	-	10	_
	ECA-M01	91	5	4	-	-	1
	ECA-M03			12	-	-	3
	ECA-M05			20	-	-	5
	ECA-M10			40	-	-	10

ECP EVA/clay predispersion, LAP Laponite XLG, CSN Cloisite Na+, ME S1ME

at 100 °C. The procedures for the preparation of the EVA/ clay adhesive sheets are illustrated in Fig. 3.

2.3 Characterization

2.3.1 Zetasizer

The particle size distribution and zeta potential were investigated via dynamic light scattering (DLS) at 25 °C using a Zetasizer Nano ZS apparatus from Malvern Instruments, Ltd., UK. The clay (0.2 g) was added to deionized water (200 g) and mixed. The suspension was ultrasonicated at a frequency of 20 kHz for 1 min at room temperature by an ultrasonic processor from Sonic & Materials, Inc., USA.

2.3.2 X-ray diffraction (XRD)

Wide-angle X-ray scattering analysis was performed to confirm the organically modified clay and the clay dispersion in the adhesive sheets using a Bruker X-ray diffractometer (equipped with a 2-D detector) in reflection mode. The tests were carried out with 2θ scanned between 2.0° and 10° by nickel-filtered Cu K_{al} radiation (k=0.15418 nm) at a voltage of 40 kV and a current of 30 mA.

2.3.3 Thermogravimetric analysis (TGA)

The TGA measurements were acquired using a thermogravimetric analyzer (TGA 4000, Perkin Elmer Life and Analytical Sciences, USA) with samples having a mass from 5 to 7 mg. The samples were loaded into a ceramic pan and run three times for each sample to confirm the reproducibility. The heating rate was 10 °C/min over the temperature range from 60 to 600 °C, and a nitrogen atmosphere was employed.

2.3.4 Cone calorimetric test (CCT)

The flame retardancy was assessed by a cone calorimeter (Fire Testing Technology LTD., UK) according to the ISO 5660-1 standard. First, the back side of the specimens (100 mm \times 100 mm \times 4 mm, W \times L \times T) was covered with aluminum foil and covered with a metal cover, after being loaded into the metal holder, and irradiated horizontally at a heat flux of 50 kW/m².

3 Results and discussion

3.1 Zetasizer

Figure 4 shows the particle size distributions and zeta potentials that were calculated using dynamic light scattering (DLS). The particle size of LAP was approximately 500 nm, and those of CSN and ME were similar to each other, at approximately 1200–1300 nm. However, CSN has a larger standard deviation than ME, which means that the size distribution is much larger than that of ME. In the case of LAP, there was a tendency to show two peaks, while CSN and ME showed only one peak. This finding is attributed to the agglomeration of the smaller nanosized particles of



Fig. 3 Ideal process for the preparation of EVA/clay adhesive sheets



Fig. 4 Particle size and zeta potential of clays in DI water

LAP rather than the others. This trend of the initial specific capacity decreasing with the particle size is inconsistent with other published studies and can be attributed to the increased aggregation in small nanoparticles (Cen et al. 2017; Liu et al. 2005). Additionally, the zeta potential result is presented in Fig. 4. The zeta potential measures the colloidal stability of dispersed systems due to the dispersibility of different nanosized particles. All values of the clays exceeded 30 mV, and their values were similar. In previous studies, dispersions with a zeta potential above 30 mV were considered to be electrostatically stable (Vo and Plank 2018; Somasundaran 2006). From these results, all suspensions were confirmed as electrostatically stable, but the LAP, of approximately 500 nm, showed a partial flocculation phenomenon.

3.2 X-ray diffraction (XRD)

The organizational degree of clay can generally be confirmed by changing the interlayer spacing using XRD. Tian et al. (2004) organically modified the montmorillonite (MMT) with octadecylammonium (ODA) and then compared the interlayer change between pristine MMT and the organically modified MMT (OMMT) using XRD. The interlayer spacing of the pristine MMT and OMMT was 1.50 and 2.01 nm, respectively. The interlayer spacing of the OMMT increased due to the ODA intercalating into the galleries of silicate layers. The XRD patterns of the pristine clays and organically modified clays in the range of $2\theta = 2-10^{\circ}$ are presented in Fig. 5. As seen in the graph, the interlayer spacing between the clay platelets increased as the organic modification occurred. This behavior was particularly confirmed when the particle size of the clay was slightly larger. These observations agreed with similar results from previous studies (Khodaeimenhr et al. 2018; Oueslati et al. 2009; Bruna et al. 2012). The modified LAP with CTAB changed the interlayer spacing from 1.4 nm to 1.5 nm. The organic modification also increased the interlayer spacing of CSN from 1.2 to 1.9 nm. In addition, the organically modified ME showed interlayer spacing changes from 1.0 to 1.5 nm and 2.2 nm. From these results, it was confirmed that the interlayer spacing changes according to the particle size for the same concentration in the organic modification. As shown in Fig. 6, the interlayer spacing change of ME exhibits the highest value (1.2 nm) among the clays, and that of the LAP, with the lowest particle size, presents the lowest value (0.2 nm).

Figure 7 shows the XRD patterns of the EVA/clay adhesive (ECA) sheets as a function of the organically modified clay content. The pattern of ECA with a clay content of 1 phr revealed that each clay peak was absent, which is evidence that the clay was intercalated. The clay piles were spread out by the polymer chains, and then the clay platelets were dispersed randomly, which caused the XRD intensity to disappear or broaden. In many previous studies, the intercalation of clays generated lower clay contents (Akin and Tihminlioglu 2018; Cervantes-Uc et al. 2007; Gatos and Karger-Kocsis 2007). However, the XRD reflection started to increase above 3 phr of the clays and then increased consistently with the clay content due to the generated agglomeration of the clays. The aggregation occurred due to the overloading of the amount of clay, and the phenomenon of aggregation increased continuously with the amount.

3.3 Thermal properties

The TGA measurement results of the ECA sheets are shown in Figs. 8 and 9. Figure 8 provides the TGA curves for both the neat EVA and ECA sheets. Regardless of the size, the final residue of all samples increased continuously with the clay content. These results have also been confirmed in previous studies (Tsai et al. 2016). However, interestingly, in the case of ME, as the clay content increases, the pyrolysis temperature continuously increases over a range from approximately 300–600 °C, whereas LAP and CSN show no trend for the same section. From this result, it can be seen that the degree of organic modification is not uniform according to the size of the clay, and the additive does not exhibit a constant thermal stability.

The thermal properties were investigated by measuring the temperature at 95% and 50% residual mass losses and the amount of final residue, depending on the type of clay (Fig. 9). The temperatures at mass losses of 95% and 50% represent thermal stability and heat resistance, respectively. The graph confirmed that there is no relationship between the size and the content of the clay, and the samples have similar values when the mass loss is 95% and 50%. Since clay can be obtained from natural rocks or soil, it does not cause any chemical reaction at the time of combustion and changing thermal properties. Conversely, the



Fig. 5 XRD patterns of pristine clays and organic-modified clays: a LAP, b CSN, and c ME



Fig.6 Interlayer spacing from XRD results as a function of the organic modification

final residue showed a tendency to increase continuously with the clay content. Clay, as mentioned above, does not undergo a chemical reaction and physically blocks the energy generated during combustion, thereby acting as a flame retardant; therefore, increasing this amount enhances the flame retardancy. However, in the case of the final residue according to size, a big difference of 5 phr was evidenced. In the case of ECA-L series, the sample showed a larger final residue value than did ECA-M series and ECA-C series (You can see in Table 1). This result is because of the increased extent to which EVA-M and ECA-C, which are large in size, were organically modified, and the sample contains an abundance of burned substances per content. Therefore, the results from the XRD data in Fig. 5 can be confirmed. In addition, in the case of ME compared to CSN, which is generally widely used, the content of 10 phr indicated a slightly higher final residual content.



Fig. 7 XRD patterns of ECA as a function of the organic-modified clay content: a ECA-L, b ECA-C, and c ECA-M

3.4 Flame retardancy

To measure the flame retardancy of the materials, the most widely used calorimeter was used. Regardless of the type of clay, all ECA series showed a tendency to decrease the heat release rate (HRR) as the clay content increased (Fig. 10A). However, it was confirmed that as the clay content increases, the HRR decreases greatly in the case of CSN and ME, which are large in size relative to LAP. Compared with the TGA results, the final residue of the sample with LAP (5 phr) was considerably higher than that of the samples with CSN and ME. However, in the case of the HRR obtained from the cone calorimeter test, the opposite result occurred. As described above, since there is no chemical reaction, and the clay physically blocks the energy generated during combustion (thus exhibiting barrier properties), even when the final residual amount is smaller, the larger sized clay showed a better flame retardancy than did the smaller sized clay. For these reasons, when the peak heat release rate (PHRR) values were compared, the EVA sheets prepared with larger sized clay showed far lower PHRRs than those prepared with smaller sized clay. Figure 10A also shows that the peak heat release rate time point tended to be faster than that in the neat EVA. This phenomenon shows that the EVA molecules undergo degradation due to deacetylation under the high-temperature conditions of the extrusion process for EVA/ clay adhesives, thereby decreasing the molecular weight or generating double-bond internal structures in EVA (Nyambo et al. 2009).

Figure 10B shows the average rate of heat emission (AHRE) curves. This parameter represents the accumulated heat emissions divided by the time and has been proposed for investigating the tendency of fires in actual environments. The maximum average rate of heat emission (MAHRE) is also an important indicator to confirm the real combustion trend (Wu et al. 2010). In general, the MAHRE value of ECA-L shows no significant difference as the content increased; however, the value did decrease by only approximately 2% with content up to 10 phr. On the other hand, the MAHRE values of ECA-C and ECA-M decreased continuously as the clay content increased, and the MAHREs decreased by approximately 44% and 34%, respectively. From these results, it was confirmed that the particle size and the size distribution of the clay influence



Fig.8 TGA curves of ECA as a function of the organic-modified clay content: a ECA-L, b ECA-C, and c ECA-M

the AHRE. In addition, when the particle size is relatively large and the size distribution is uniform, the MAHRE value is smaller.

Smoke and toxic gases from fires are also meaningful factors in fire safety. These factors were observed because smoke has the potential to delay people's escape, and toxic gas can cause injuries (Wu et al. 2010). Figure 10C reports the CO production rate of the ECA series as a function of the clay. The CO production of ECA-L decreased by approximately 20%; however, there was no significant difference according to the content of LAP, and the end point of CO generation was slightly delayed. While the values of ECA-C and ECA-M dramatically decreased as the clay content increased, the endpoint of the CO production rate was consistently delayed. The delay of the endpoint means that the escape time is longer, which is closely related to fire safety. Figure 10D shows the smoke production rate (SPR) of the ECAs during combustion. The SPR value of ECA-L was approximately 8% higher than that of the neat EVA. From these results, it can be seen that the LAP exhibits a barrier-like property to reduce the HRR to a small extent, but clay with a too small particle size increases the SPR value. Conversely, the values of ECA-C and ECA-M significantly decreased at approximately 18% and 26%, respectively. In addition, it was confirmed that the endpoint of SPR was delayed as the clay content increased. In other words, the larger the particle size is, the more favorable the CO generation rate and the SPR. Furthermore, the size distribution showed no significant difference with respect to the particle size, but the homogeneous particle distribution was more effective.

During the combustion, the EVA/clay sheets turned into a multilayered carbonaceous-silicate structure. This structure is formed due to the EVA polymer matrix that is decomposed by radiant heat and due to the clay particles that remain (Beyer 2002; Jeffrey 1999; Lexzczynska et al. 2007a, 2007b; Kiliaris and Papaspyrides 2010). The carbonaceous char produced on the surface during combustion was enhanced by silicate, thereby creating an excellent physical barrier that blocks the heat and oxygen and retards the release of flammable volatiles generated by polymer degradation (Jeffrey 1999; Alexandre and Dubois 2000; Zanetti et al. 2004; Wang et al. 2002; Du et al. 2002). Figure 11a shows the neat EVA before and after combustion acquired using a cone calorimeter. As expected, the adhesive sheet formed of pure EVA polymer



Fig. 9 TGA results of ECA as a function of the organic-modified clay content: a ECA-L, b ECA-C, and c ECA-M

was volatilized completely and without residue during combustion. When comparing the photographs of carbonaceous char formed after the burning of EVA sheets with the same clay content, it was confirmed that a strong char layer was formed with CSN and ME (Fig. 11b).

Figure 12a, b shows the ideal mechanisms for organically modified clays and the flame retardancy of the ECAs, respectively. Figure 12a suggests the difference in the organizational degree depends on the particle size. For CSN and ME, with a larger particle size, the organizational degree of the treatment is greater than that of LAP, with a smaller particle size, which can be understood by examining the interlayer spacing changes provided by the XRD results in Fig. 5. Furthermore, Fig. 9 shows the thermal properties of ECA with 5 phr of clay content. In these results, the final residue of LAP is higher than that of ME and SCN due to the larger size of clay having a greater degree of organic modification. Figure 12b shows the ideal mechanism as a function of the particle size of clay in the flame retardancy of ECAs. In general, the clay exhibits flame retardancy by physically shielding the energy generated during combustion without being chemically reactive. Therefore, the larger the particle size is, the better the flame retardancy due to enhancement in the barrier properties.



Fig. 10 Cone calorimeter test results of ECA as a function of the organic-modified clay content: A HRR, B AHRE, C CO production rate, and D SPR. a ECA-L, b ECA-C, and c ECA-M



Fig. 11 Char layers after combustion: a Neat EVA, and b EVA/clay sheet with 10 phr of clay



Fig. 12 Ideal mechanisms as a function of clay: a organic modification of clays, and b the flame retardancy of ECAs

4 Conclusion

From the Zetasizer results, it was confirmed that the clays have different particle sizes and distributions and have zeta potential values above 30 mV. The XRD results of all clays showed that the interlayer spacing between the clay layers increased. Among the clays, ME-CTAB showed the greatest change. This result means that the size distribution of the clays could affect the degree of organic modification under the same conditions. When the organic-modified clays are blended with EVA, the XRD results showed a broad curve due to the clays being well dispersed in the EVA matrix. According to the TGA test, the final residue of all samples increased with the clay content. However, only the ME sample showed a continuous thermal property change during the temperature sweep. In addition, the final residue of LAP with 5 phr is considerably higher than that of CSN and ME. The particle size and size distribution are confirmed to affect the thermal properties. Regarding the cone calorimeter test, the HRR decreased with the clay content. Notably, CSN and ME, which have considerably larger sizes, were more effective in lowering the HRR. From these results, the clay sizes and size distributions are important factors in determining the properties of EVA/ clay adhesive sheets. In addition, it was confirmed that the EVA/clay adhesive sheets have potential uses as adhesive materials for CLT. Therefore, in a future study, the authors will prepare the CLT with EVA/clay adhesion sheets to enhance the flame retardancy of CLT and investigate the adhesion performances.

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