

Fire Performance of Fiber Board Coated with Nano Kaolin-Clay Film

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Fiberboard is a common interior material used both in China and the United States of America. The increase in demand for interior materials has raised concerns regarding combustibility of the materials. The pyrolysis characteristics of fiber, phenolic resin (PF), and nano kaolin-clay (NK) were investigated using thermogravimetry. The fire performances of samples coated with a mixture of NK and PF were determined using a cone calorimeter. The pyrolysis process of fiber included three phases dependent on chemical composition. The initial temperature of PF pyrolysis was about 100 °C and it stopped at 280°C. The major mass loss of NK was observed between 400 to 600 °C due to the gradual oxidation of the metakaolin. In comparison with fiber board, samples coated with a mixture of NK and PF achieved a better fire performance. The results showed a longer TTI, lower HRR, and THR, and less CO and CO₂ yield, especially from a mixture of NK (90%) and PF (10%). The application of an NK and PF mixture to fiber board as a flame retardant is an effective method for enhancing fire safety and resistance.

Keywords: Fire performance; Fiberboard; Recycled fiber; Kaolin clay; Phenolic resin

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INTRODUCTION

Kaolin [Al₂Si₂O₅(OH)₄] clay is a common phyllosilicate mineral that belongs to a large general group of clays (Wang *et al.* 2011). Its structure is composed of silicate sheets (Si₂O₅) bonded to aluminum oxide/hydroxide [Al₂(OH)₄] layers. Kaolin is used as a raw material for ceramics, porcelain, paper, coatings, rubber, plastics, fire-proof materials, chemicals, pesticides, medicines, textiles, petroleum, and building materials. The literature shows a variety of composites that have been modified using kaolin-clay. Blow-molding grade high-density polyethylene filled with kaolin has been characterized by wide-angle X-ray scattering, microhardness, and stretching calorimetry techniques. It was proven that crystallinity of the polymer matrix in the filled samples remained essentially the same as that in the virgin polymer regardless of the filler content (Privalko *et al.* 1999).

The crystallization behavior of kaolin-filled polypropylene composites with various kaolin content, processing temperatures, and shear histories has also been investigated by differential scanning calorimetry. It was found that increasing kaolin content, processing temperature, and shear stress shifted the crystallization exothermic peak to a higher temperature and reduced the overall crystallization time (Jikan *et al.* 2010). The adsorption behavior of surfactants (hexadecylpyridinium bromide, sodium dodecylbenzene sulfonate, and Triton X-100) onto kaolin from an aqueous solution was

monitored as a function of pH. The nature and shape of the adsorption isotherms were typically very much dependent on the surfactant structure. All three surfactants adsorbed onto kaolin beyond the limits of a monolayer coverage (Sastry and Dave 1999).

Low-cost tile clay mixed with kaolin has been evaluated by some thermal analysis methods. The initial melting temperature was observed to increase upon kaolin addition (Albuquerque *et al.* 2004). Packed bed sorption experiments for the capture of gaseous cadmium and lead using calcined kaolin particles were performed in a temperature range of 973 to 1173 K. Cadmium and lead reacted with the sorbent to form water-insoluble metal-mineral complexes. An increase in bed temperature resulted in an increase of capturing rates, but had no effect on maximum uptakes for either metal.

Wood-based composites are used for a number of structural and nonstructural applications including panel products (plywood, oriented strandboard, particleboard, fiberboard, medium-density fiberboard, hardboard), structural timber products (glued-laminated timber, laminated veneer lumber, laminated strand lumber, parallel strand lumber) and wood-nonwood composites (wood fiber-thermoplastics, inorganic-bonded composites) (Cai and Ross 2010).

Fiberboard is a traditional type of wood-based composite and is primarily manufactured using high-yield thermomechanical pulp, forest residuals, and recycled fibers (Hunt *et al.* 2008). Fiberboard can be classified into the following categories based on density: low-density (200 to 400 kg m⁻³) fiberboard, medium-density (400 to 800 kg m⁻³) fiberboard, and high-density (> 800 kg m⁻³) fiberboard. Kang (1998,2000) measured cushioning performance of pulp-molded products and correlated it to design variables that included unit height, bottom-unit perimeter length, unit-slope angle, radius of the comers, shape-factor coefficients, and pulp thickness. Hunt (1998) showed that the physical and mechanical properties of wet-formed flat sheets made from several types of recycled fibers including old newspapers and old corrugated containers (OCC) are possible and show similar properties as commercial hardboard. Suchsland *et al.* (1998) also showed that recycled newsprint and kraft fiber could be substituted into the hardboard process by means of a modified wet-formed process. Hunt and Vick (1999) demonstrated that high strength panels could be produced from recycled OCC with the wet-formed technology when the panels were press-dried between two screens. Hunt and Supan (2005, 2006) examined the effects of varying moisture content on performance characteristics. It was found that strength and stiffness decreased with increasing amounts of moisture.

While there are numerous papers and patents that describe clay coatings as a fire retardant, only a few have considered the effect of the interaction between clay and phenol-formaldehyde on fire retardant performance (Liu and Zhu 1999). This paper discusses an initial investigation using nano kaolin (NK) on the surface of high-density fiberboard that was manufactured using OCC by a wet-form process. The purpose of the research was to investigate the effect of NK on fire performances of fiberboard. The panels were coated with a mixture of NK and PF at different mass ratios. The pyrolysis characteristics of fiber, PF, and NK were investigated using thermogravimetry. The fire performances of samples coated by a mixture of NK and PF were determined using a cone calorimeter.

MATERIALS AND METHODS

Materials

The fiberboard panels were made using a binderless wet-formed process (Hunt and Supan 2006). The OCC (a mix of corrugated box trimmings obtained from a local box plant) was hydropulped at the Forest Products Laboratory (FPL) to an approximate freeness of 600 CSF. The pulp was wet-formed and hot-pressed to produce nominal 2.5 mm thick fiberboard. Binderless panels made from the OCC pulp fibers had very good mechanical properties (Hunt *et al.* 2008). The NK was obtained from IMERYS Pigments & Additives Group, USA. Its composition included 50 to 60% kaolin clay, 40 to 50% water, <0.1% to <0.3% crystalline silica, <0.4% sodium polyacry/soda ash, <0.2% FDA approved suspension aid, and <0.1 % FDA approved biocide.

The phenolic resin was a commercial phenol-formaldehyde resin with approximately 50% solids.

The fiberboards were manufactured by a wet-forming process at 1 % consistency [consistency=dry fiber weight/ (water + fiber weight)]. After forming and vacuum pressing, the sheets had approximately 75% moisture content (MC) on a wet basis (300% MC dry basis). Each mat was placed between two screens and two 3.2 mm thick stainless steel cauls. The wet mats were hot pressed at 180 °C with continuous pressure (483 kPa) to target densities of 850 kg m⁻³ until the panels were dried (approximately 1% to 2% MC) (Hunt *et al.* 2008). Final board thicknesses were between 2.7 to 2.9 mm thick.

After fiberboard fabrication, the fiberboard surfaces were coated using a series of mixtures of NK and phenol-formaldehyde (PF) with the ratios of 100:0, 95:5, 90:10, 80:20, 50:50, and 0:100, respectively. About 5% (w/w) of the mixture was put on the surface of each sample. The coating was applied by hand using a wide blade scraper. A specific amount was weighed out and then spread over the fiberboard surface. Coating thicknesses ranged from 0.24 to 0.25 mm with a combined average density of between 1000 and 1150 kg/m³. Total coating thickness represented approximately 15% total thickness or 7.5% of the total thickness per each side. All samples were hot pressed at 160 °C for 30 s to dry the board and clay as well as cure the PF. Minimal pressure was used to allow the moisture to escape from the coating to help maintain the boards to remain flat during drying. Samples (100 mm × 100 mm) were obtained from each panel and placed into a conditioning room set at a temperature of 27 °C and relative humidity of 65% for two weeks. Panel equilibrium MC was 8%. Finally, all samples were transferred to separate Ziploc bags and sealed tightly prior to testing.

Property Tests

Thermal decomposition

Materials for the thermal decomposition test included fiber, PF, NK, and mixtures of NK and PF that were first broken down into particles using a Wiley Mill. These materials were then screened to get 250 to 425 μm particles. About 100 g particles were dried at 105 °C until the mass variance of the crucible was less than 0.2%.

Thermal decomposition of all materials was measured in terms of total mass loss through a TA Instrument TGA Q 500 thermogravimetric analyzer (TA Instrument, USA). First, the different types of particles were evenly and loosely put into an open sample pan; the initial sample weight was about 3 to 6 mg. The temperature change was controlled from 30 ± 3 °C to 900 °C with a heating rate of 20 °C /min. High purity nitrogen gas with a flow rate of 60 mL/min was continuously passed into the furnace

before thermal decomposition was carried out in order to prevent any unwanted oxidative decomposition. The experimental data could be directly obtained through a TGA Q 500 thermogravimetric analyzer, but they were analyzed using Universal Analysis software from TA Instruments and Origin 8.0 software.

Fire performance

Fiberboards with surface coatings of NK and PF mixtures were tested using a cone calorimeter (Fire Testing Technology LTD., UK) with ISO 5660-1 standard method. The tests were done at the horizontal orientation. The data obtained were: heat release rate (the heat evolved from the specimen, expressed per unit of exposed specimen area, per unit of time; kW/m²); total heat release (total heat generated by the sample over the entire test period, calculated by integrating the curve of heat release rate vs. time; MJ/m²); mass-loss rate (mass lost per time; g/s); carbon monoxide production rate (g of CO/s); carbon dioxide yield (yield of CO₂ averaged over the entire test period, based on mass of sample consumed; kg of CO₂/kg of material), and time to ignition (determined visually and taken to be the period required for the entire surface of the sample to burn with a sustained luminous flame) (Ivan *et al.* 2007). All samples were tested at the 35 kW/m² flux, using an edge frame. Three identical samples were used to determine repeatability.

RESULTS AND DISCUSSION

Thermal Decomposition

Figure 1 shows the pyrolysis process of fiber at a heating rate of 20 °C /min. The pyrolysis process for fiber has three distinct stages similar to those observed in a previous study for wood materials (Yao *et al.* 2008). The initial temperature for each stage is defined as the base point for mass change at that stage. For the next stage, the final temperature of thermal decomposition for the previous step becomes the initial temperature for thermal decomposition. In the third stage, the final temperature is defined as the critical point where the mass variance of sample is very slight (Jiang *et al.* 2011).

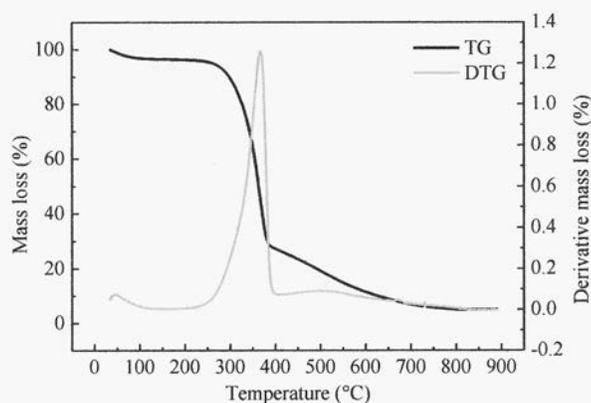


Fig. 1. Pyrolysis process of fiber at a heating rate of 20 °C /min

In the first stage, the degradation temperature was 30 °C to 100 °C, and mass loss was due to removal of water from the samples. In the second stage, the degradation temperature was 100 °C to 380 °C, and the degradation of cellulose, hemicelluloses, and

partial lignin accounted for the weight loss during this stage. The critical temperature for maximum mass loss was 365 °C for fiber in the pyrolysis process. In the third stage, the degradation temperature was 380 °C to 800 °C, and weight loss was attributed to the degradation of lignin residues from the second stage or tar and char from the main components.

Figure 2 shows the pyrolysis for PF at a heating rate of 20 °C /min. The initial temperature of PF pyrolysis was about 100 °C, and it stopped at 280 °C. The mass loss of PF was due to some pyrolysis products. Serio *et al.* (1998) found that the pyrolysis products included CO, CO₂, CH₄, H₂O, SO₂, C₂H₄, and NH₃ when the pyrolysis temperature was lower than 250 °C. Aliphatic, aromatic, hydroxyl, carbonyl, and ether groups were evolved in the thermal composition process above 250 °C.

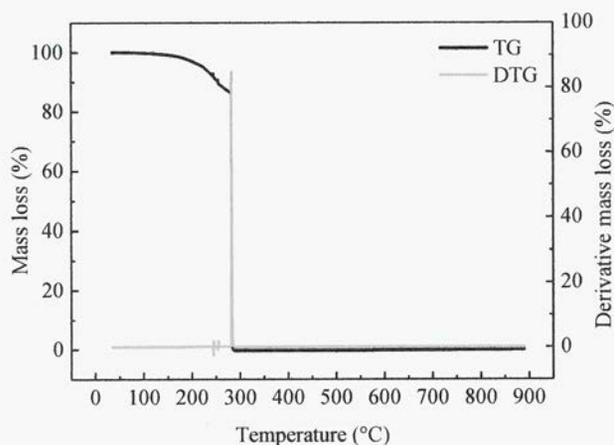


Fig. 2. Pyrolysis process of PF at a heating rate of 20 °C /min

The TG curves for mixtures of NK and PF samples between 30 to 900 °C are shown in Fig. 3. The major mass loss for the NK sample was observed between 400 and 600 °C. This may be due to the gradual oxidation of the metakaolin. Metakaolin is not a simple mixture of amorphous silica (SiO₂) and alumina (Al₂O₃), but rather a complex amorphous structure that retains some longer-range order (not strictly crystalline) due to the stacking of its hexagonal layers (White *et al.* 2010).

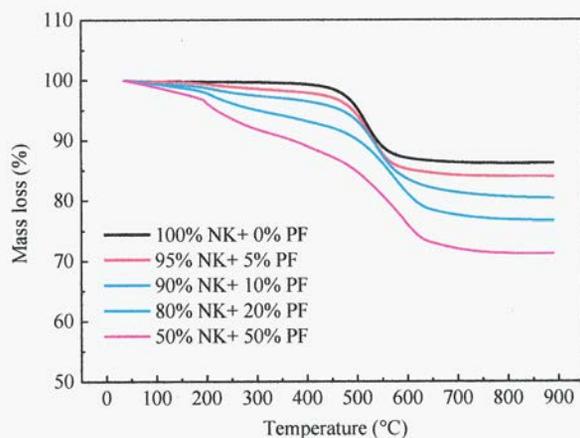


Fig. 3. The mass loss of NK or mixture of NK and PF in the pyrolysis process

The structure loses its crystallographic order between 500 and 750 °C, which is observed by the lack of Bragg scattering. The minimum mass loss occurred when pyrolysis temperature was below 400 °C or above 600 °C; this can be attributed to endothermic dehydroxylation (or alternatively, dehydration) to produce disordered metakaolin ($\text{Al}_2\text{Si}_2\text{O}_7$). Dehydroxylation continued up to 900 °C (Wang *et al.* 2011). The general feature for mixtures of NK and PF samples was similar to those observed in NK samples. As PF content increased, there was a greater mass loss in the samples of NK and PF mixtures. PF could be responsible for this phenomenon, which was also confirmed by the results from Fig. 4. There were two maximum mass loss peaks for samples of NK and PF mixtures. The first peak located at the lower temperature indicated the mass loss for PF while the higher peak located at the higher temperature resulted from the mass loss for kaolin clay.

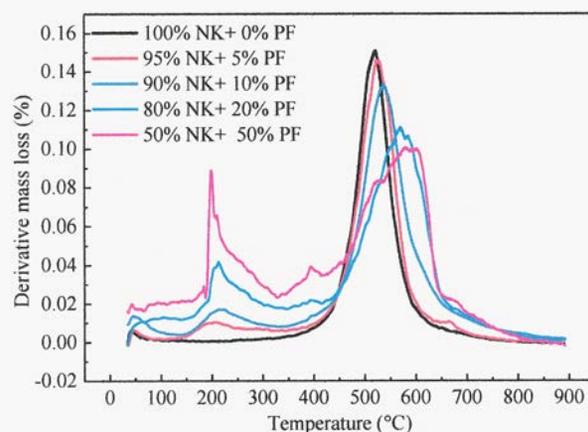


Fig. 4. The derivative mass loss of NK or mixture of NK and PF in the pyrolysis process

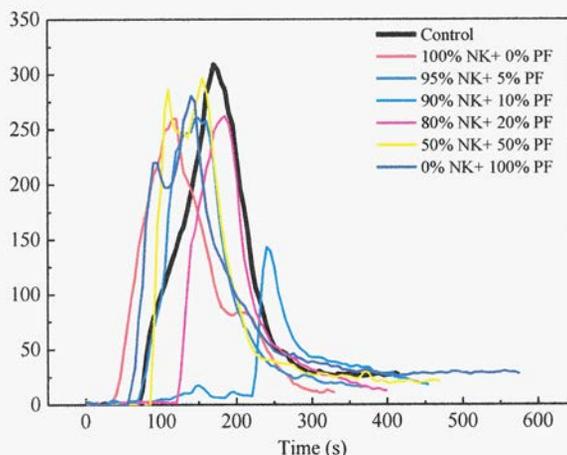
Fire Performance

Time-to-ignition (TTI) is the elapsed time required to produce ignition under thermal radiation. This measurement is an important parameter to assess the fire risk of materials. A shorter TTI means it is easier for the materials to be ignited and that they are at greater risk of catching fire. Table 1 shows the TTI for fiberboard and fiberboard coated with different mixtures of NK and PF at the heat flux of 35 kW/m². Samples coated with NK and PF promoted a faster TTI compared with fiberboard control samples. At higher concentrations of PF there are some combustible gases such as CH_4 , C_2H_4 , and NH_3 in the PF pyrolysis, which may have promoted the samples coated by PF to ignite sooner. NK is a type of phyllosilicate mineral, which is composed of silicate sheets (Si_2O_5) bonded to aluminum oxide/hydroxide [$\text{Al}_2(\text{OH})_4$] layers (Wang *et al.* 2011). NK has a very good thermal conductivity and thermal stability (Yang *et al.* 2001), but it was observed that the NK was poorly bonded to the surface of the fiberboard. The 100% NK-coated fiberboard exhibited random cracks on the panel surface due to the differential thermal stability between NK and the fiberboard during the test. It is possible that the volatile gases were concentrated in the cracks, causing faster ignition. When PF was added to the NK, the NK-fiberboard bond was observed to improve. It was very obvious that TTI increased for samples coated with a mixture of NK and PF and reached a maximum when PF was mixed in at 10%.

Table 1. Time to Ignition for NK-Coated Fiberboard Samples

Sample Types	Control	100%NK+ 0% PF	95%NK+ 5% PF	90%NK+ 10% PF	80%NK+ 20% PF	50%NK+ 50% PF	0%NK+ 100% PF
TTI (s)	71	38	68	220	121	83	54

It is well known that combining TTI, heat release rate (HRR), and total heat release (THR) could make a better assessment of fire risk. HRR and TTI are consistent with exterior heat flux, ventilation degree, and destruction degree of materials. THR indicates the interior energy of materials, which is independent of environmental factors. The fire risk of materials is better predicted by combining HRR, TTI, and THR and is closely related to the results of large-scale experiments (Lee *et al.* 2011). Figure 5 shows HRR results for the samples. For the 90:10 NK:PF fiberboard, there was an initial peak in the HRR curve due to thermal decomposition of products on the surface of the samples. As the samples char, the heat release rate gradually drops to a somewhat steady-state rate. The general feature of HRR variation with time was similar to those observed in a previous study of wood materials (Winandy *et al.* 2008). HRR is an important parameter to describe materials as a fire hazard. A higher HRR value means that the material makes more heat and affects other materials to increase the pyrolysis rate. At a high pyrolysis rate, flame spread is promoted, because more volatile combustible gases are released (Babrauskas and Peacock 1992). The HRR value for the samples coated with a 90:10 NK:PF mixture was the lowest of the samples. The mass loss rate of the samples was also determined by cone calorimeter test and is showed in Fig. 6. The mass-rate-losses for the samples were similar to those for HRR. THR process during the test included three phases (Fig. 7). The first phase ended at TTI for the samples and was related to initial surface degradation. The THR rapidly increased after the samples ignited. The second phase ended at flameout. During combustion, a char layer from pyrolysis of the main components was formed on the surface of samples and THR started to slow. It was also obvious that the THR of samples coated with a mixture of NK (90%) and PF (10%) was lower than that of any other samples. The results from this research suggest greater safety for samples coated with a mixture of NK (90%) and PF (10%).

**Fig. 5.** The heat release rate of samples

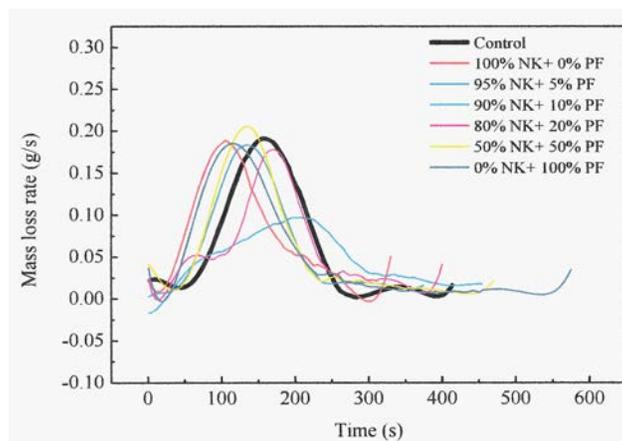


Fig. 6. The mass loss rate of samples

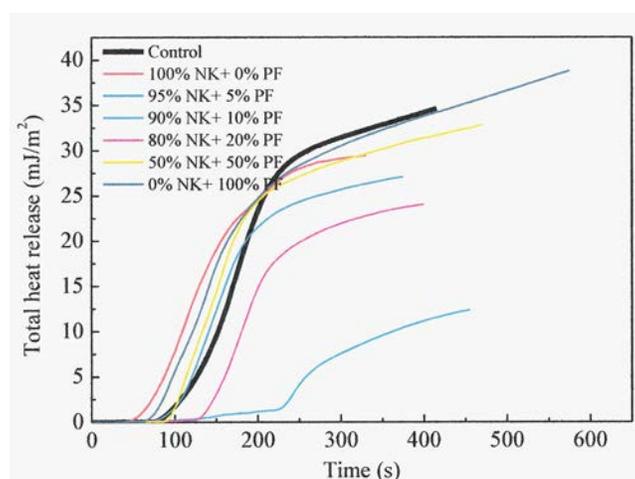


Fig. 7. The total heat release of samples

Most materials release dense smoke in the combustion process. Smoke limits visibility and can cause disorientation for people attempting to escape from a fire. The production of smoke and toxic gas plays an important role in understanding the fire hazard related to interior materials (Mouritz *et al.* 2006).

Figures 8 and 9 show the CO and CO₂ yield of samples. Fiberboard is composed of cellulose, hemicelluloses, lignin, and a remaining smaller part comprising of extractives and minerals. The gas products were released due to thermal decomposition of materials and depended on the chemical composition, oxygen availability, and fire temperature (Mouritz *et al.* 2006). After an ignition time of 300 s, CO and CO₂ were emitted from samples. The CO and CO₂ yield of fiberboard was higher than that of samples coated with mixtures of NK and PF. This was probably due to the fire retardant property of NK and PF mixture.

It was also obvious that the CO and CO₂ yield of samples coated with a mixture NK (90%) and PF (10%) was the lowest in all types of samples. The importance of reducing smoke to increase human survival in a fire has prompted the characterization of the smoke properties for a wide range of interior and construction materials (Lee *et al.* 2011).

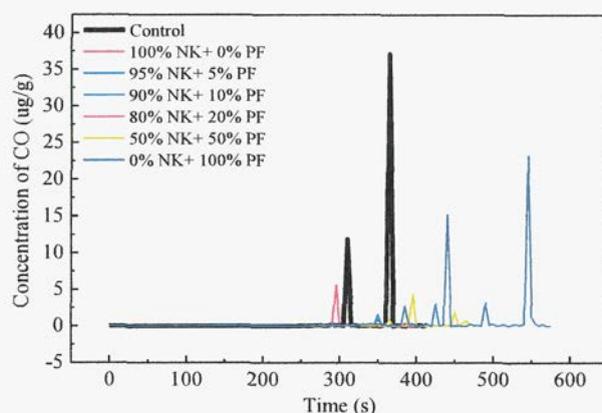


Fig. 8. The CO yield of samples

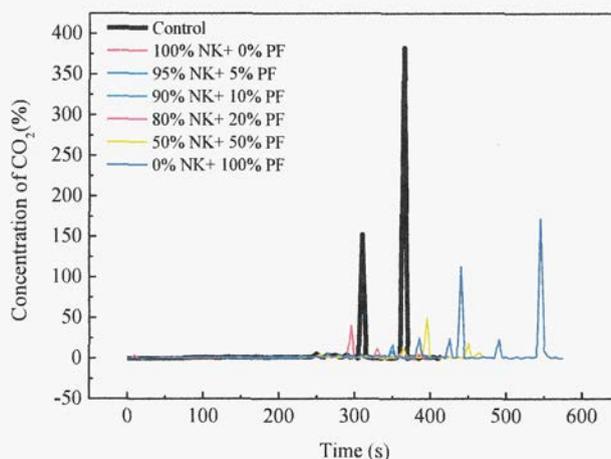


Fig. 9. The CO₂ yield of samples

CONCLUSIONS

1. The pyrolysis characteristics of fiber, phenolic resin (PF), and nanokaolin (NK) were investigated using thermogravimetry. The pyrolysis process for the fiber was similar to those observed in a previous study of wood materials.
2. The pyrolysis of PF included two phases dependent on chemical compositions. The initial temperature of PF pyrolysis was about 10 °C, and it stopped at 280 °C. The pyrolysis products included CO, CO₂, CH₄, H₂O, SO₂, C₂H₄, and NH₃ when pyrolysis temperature was lower than 250 °C. Aliphatic, aromatic, hydroxyl, carbonyl, and ether groups were evolved in the thermal composition process above 250 °C. The major mass loss from the NK sample was observed at 400 to 600 °C, due to the gradual oxidation of the metakaolin.
3. In comparison with the control sample, fiberboard with no coating, samples coated with a mixture of NK and PF achieved a better fire performance such as a longer time to ignition (TTI), lower heat release rate (HRR), and total heat release (THR), and less CO and CO₂ yield, especially with a mixture of NK (90%) and PF (10%).

4. The application of NK and PF mixture to fiber board as a flame retardant is an effective method for enhancing the fire safety and resistance.

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