

# Synthesis and Thermal Behavior of Inorganic–Organic Hybrid Geopolymer Composites

M.Hussain,<sup>1</sup> R. Varely,<sup>2</sup> Y. B.Cheng,<sup>1</sup> Z.Mathys,<sup>3</sup> G. P.Simon<sup>1</sup>

<sup>1</sup>School of Physics and Materials Engineering, Monash University, Wellington Rd, Clayton, Vic-3168, Australia

<sup>2</sup>CSIRO, Molecular Science, Clayton, Vic-3168, Australia

<sup>3</sup>DSTO, Platform Laboratory, Melbourne, Australia

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**ABSTRACT:** Inorganic geopolymer potassium aluminosilicate was prepared at room temperature by the reaction of kaolin, potassium silicate, and potassium hydroxide solution and was dispersed *in situ* into an epoxy matrix by various proportions to fabricate novel inorganic–organic hybrid geopolymer composites. The formation of inorganic geopolymer with respect to time was monitored by X-ray diffraction and FT-IR analysis and confirmed that 30 min is required to complete the geopolymerization. When geopolymers were properly mixed at different ratios with organic polymers such as epoxy and cured, these hybrid polymers exhibit significant thermal stability. Pure kaolin was also incorporated into the epoxy matrix to compare the change in chemical and thermal properties. Cone calorimetry results showed about 27% decreased in rate of heat release (RHR)

on addition of 20% pure kaolin. However, about 57% of RHR was decreased on addition of only 20% geopolymer. Evaluation of CO<sub>2</sub> and CO were found to be minimum 2.0 and 0.7 kg/kg, respectively, for hybrid geopolymer composites compared to very high yield for epoxy at 3.5 kg/kg after 200 s of ignition. The current study shows that due to the high thermal stability of hybrid geopolymer composites, the novel hybrid geopolymer composites have the ability to be potential candidates to use in practical application where fire is of great concern. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 96: 112–121, 2005

**Key words:** geopolymer; thermal properties; epoxy; hybrid calorimetry; cone calorimetry

## INTRODUCTION

Many advanced polymer composites consist of an organic phase of highly crosslinked resins, often from the epoxy family. The fusible, soluble starting materials can incorporate various fillers, such as glass, liquid crystal polymer (Kevlar) fibers, carbon fibers, ceramic particulates, and clays, in a variety of ways to improve the final physical, mechanical, and thermal properties. However, the flammability of the virgin polymer matrix or additive modified polymer limits the use of these materials in many applications, such as marine platforms and ships, automobiles, and military and commercial spacecrafts, where fire hazard is an important design consideration<sup>1–3</sup> since most of the organic polymers soften and ignite at temperatures of 200–500°C, which are characteristic of fuel fire exposure conditions. Carbon fibers and glass fibers are inherently fire resistant, and significant progress has been made in recent years to develop new, high-temperature, thermo-oxidatively stable fibers from boron, silicon carbide, and ceramics.<sup>4</sup> However, these are complex and costly to make. Moreover, the presence

of the organic phase in fiber-reinforced composites will collapse or degrade at higher temperature, destroying the total structure.

Various methods have been proposed for forming inorganic protective layers on the surface of burning polymers, but their positive effect is mostly accompanied by certain disadvantages. Inorganic filler particles act through a dilution effect and reduced heat feedback. Silica additives of high surface area are most efficient as these particles accumulate on the surface instead of sinking into the polymer melt.<sup>5</sup> The physical network formed by such additives in the polymer melt may reduce dripping but, on the other hand, the same effect significantly restricts the processability of such systems. Similar problems may occur when porous fillers like zeolites are used in higher concentration, which allows the polymer chains to penetrate. At lower concentration, zeolites can be combined advantageously with intumescent flame retardant additives.<sup>6</sup>

A current area of much interest involves polymer-clay (layered silica) nanocomposites, which, when introduced in intercalated or exfoliated (delaminate) within the matrix, upon burning, may accumulate on the surface of the polymer during burning and form a barrier layer to either outgoing degradation products or incoming gases. Their effect is clearly indicated by the reduced rate of heat release of horizontal samples

Correspondence to: M. Hussain.

in the cone calorimeter.<sup>7</sup> Recent work also suggests that even very low levels (1–4%) of nano-clay lower the peak heat release rate in a cone-calorimeter by about 30%.

In addition, other strategies, such as the use of polymer precursors, which lead to formation of a protective ceramic coating in the presence of fire, have been proposed by Anna,<sup>8</sup> and are able to improve the thermal stability and fire performance. Although such systems have been examined in a number of studies, the optimization of the composition and the mechanism has not been completed.<sup>9</sup>

Looking at the broader picture, it has been thought that new inorganic materials may increasingly replace conventional plastics. This has been proposed to lead to the reduction of world pollution resulting from by-products of plastics manufacturers as well as the use and disposal cycle of these materials. In particular, a family of new materials called geopolymers (polysilates) has emerged during the last decade as one of the most promising replacement candidates for plastics in certain applications, and even ceramics, since they have appropriate physical properties of the latter materials class.<sup>10–13</sup> The manufacture of polysilates does not create CO<sub>2</sub> emissions, they can be made from recycled mineral wastes or naturally occurring geological materials, and they provide excellent acid and fire resistant and mechanical properties. Although similar to zeolites in chemical composition, they have an amorphous microstructure. The term “geopolymer” was first used by Davidovits<sup>11–13</sup> to describe a family of mineral binders closely related to artificial zeolites.

Most waste materials, such as fly ash, slag of blast furnaces, and mine tailing, contain sufficient amounts of reactive alumina and silica that can be used as source materials for *in situ* geopolymerization reactions. The most attractive geopolymers (polysilates) are inorganic polymers made from aluminosilicates since they can be synthesized at low temperatures and have useful properties, such as high compressive strength, and are stable at temperatures up to 1300–1400°C. By changing the Si/Al ratio, it is possible to produce products with very high fire resistant properties. Polysilates are readily synthesized from natural aluminosilicates, such as kaolinite, a very abundant source of alumina and silica. The formation of geopolymeric materials follows the same route as that for most zeolites. Geopolymers are formed by the copolymerization of aluminosilicate species, which originate from the dissolution of silicon and aluminum-containing source materials at a high pH, in the presence of soluble alkali metal silicates. The current commercial use of geopolymers, compared to plastics, for example, is limited because of the complexity of processing on a large scale, high density, and problems with machining and molding, and most importantly, their brittleness.

Geopolymers are linear poly(metasilicate) with tetra-coordinate aluminate crosslinks. The geopolymerization involves the chemical reaction of aluminosilicate oxides (Al<sup>3+</sup> in iv-fold coordination) with alkali polysilicates, yielding polymeric Si-O-Al bonds; the amorphous to semicrystalline three dimensional silico-aluminate structures are of the poly(silate) type (-Si-O-Al-O-), the poly(silate-siloxo) type(-Si-O-Al-O-Si-O-), and the poly(Silate-disiloxo) type (-Si-O-Al-O-Si-O-Si-O-). The atomic ratio Si : Al in the poly(silate) structure determines the properties and application areas for which they are relevant. A low ratio Si : Al (1, 2, or 3) initiates a three-dimensional network that is very rigid. A ratio of > 3 creates a two-dimensional structure, which is flexible. However, Si : Al ratios higher than 15 result in a material with a linear polymeric nature.<sup>13</sup>

This article focuses on a different approach involving geopolymers, the use of their fire retardant phase to incorporate them into crosslinked polymeric structure systems. In so doing, we make use of the processability and properties of the crosslinked polymers (such as epoxy resins), in combination with the geopolymers, to get polymeric characters with high toughness, strength, and fire resistant materials. The first system reported involves choice of a standard, bi-functional epoxy resin diglycidyl ether of bisphenol A (DEGEBA) to be incorporated with the geopolymers. The results will be compared with physically blended kaolin (the primary source of the geopolymer) with epoxy resins to investigate the ability of geopolymers within epoxy resins to act as the fire retardant component. Incorporation of geopolymers into organic polymer systems has been demonstrated for the first time and has not been reported yet.

## EXPERIMENTAL PROCEDURES

### Materials

Kaolin (HR1-F grade) with a particle size distribution of 38.20 μm was procured from Commercial Minerals, Sydney, Australia. The potassium silicate solution was obtained from PQ Australia Pty. Ltd. Potassium silicate composition was SiO<sub>2</sub>/K<sub>2</sub>O = 2.00, SiO<sub>2</sub> = 29.3 (wt %), and K<sub>2</sub>O = 14.5 (wt %), with density 1.42 gcm<sup>-3</sup>. Table I shows the chemical composition and physical properties of kaolin. 5M KOH solution was prepared in the laboratory from KOH pellets from Bdh, Merck Pty. Ltd. The epoxy resin used in the study was diglycidyl ether of bisphenol-A (DGEBA), commercially known as DER-331, from Dow Chemical Company, Australia. The curing agent used in this experiment was a mixture of 3,5-diethyltoluene-2,4-diamine and 3,5-diethyltoluene-2,6-diamine (Etha-cure-100) obtained from Albemarle Corp., USA.

**TABLE I**  
**Typical Chemical and Physical Properties of Kaolin**

Composition/Property	Chemical Composition/units	Wt %/value
Silica	SiO <sub>2</sub>	53.3%
Magnesia	MgO	0.2%
Alumina	Al <sub>2</sub> O <sub>3</sub>	30.3%
Ferric Oxide	Fe <sub>2</sub> O <sub>3</sub>	1.2%
Lime	CaO	0.2%
Potash	K <sub>2</sub> O	0.5%
Soda	Na <sub>2</sub> O	0.2%
Titania	TiO <sub>2</sub>	2.6%
Loss on ignition	(1000°C)	11.4%
p <sup>H</sup>	(20% slurry)	8.4
Specific gravity	-	2.71
Surface area	(cm <sup>2</sup> /g)	25,300
Oil absorption	(mls/100g)	50
Bulk density	g/cm <sup>3</sup>	0.6

## Synthesis

Inorganic geopolymers were synthesized by the reaction of kaolin, potassium silicate, and potassium hydroxide solution at room temperature. Initially, the desired amount of potassium silicate was mixed with a 5M potassium hydroxide solution and then 20 g kaolin was added and mixed at different periods of time. The viscous mix was then added to a mixture of DGEBA epoxy resin and curing agent with constant stirring for 15 min. The mixture was then placed into the PTFE-coated molds and was cured at 60°C for 6 h, followed by post curing at 180°C for 2 h.

To fabricate clay-dispersed composites for comparison, 20 g of pure kaolin was mixed with the mixture of DGEBA epoxy resin and curing agent for 1 h. The mix was then placed in a PTFE-coated mold, cured at 80°C for 6 h, followed by post curing at 180°C for 2 h. The sample was then cut, ground, and polished for thermal analysis, cone calorimetry, and microstructure analysis.

## Characterization

A range of techniques were used to monitor cure, and characterize the final products. The formation of geopolymers within the epoxy matrix was characterized by X-ray diffraction analysis by Rigaku wide-angle goniometer. An acceleration voltage of 40 kV and current of 22.5 mA were applied using Ni filtered Cu K $\alpha$  radiation. Fourier transform infrared (FT-IR) was recorded on a Perkin-Elmer FT-IR spectrometer. The mechanical loss tangent ( $\tan \delta$ ), in particular the determination of the glass transition temperature (T<sub>g</sub>), of the epoxy phase of the cured samples was determined on a Rheometric scientific dynamic mechanical thermal analyzer, DMTA IV. The cured samples were clamped in a medium frame using a small center clamp in the dual cantilever mode. Frequency sweep

scans were performed from 80 to 260°C at 2°C/min using frequency 1Hz and having a strain of 1%.

Thermo gravimetric analysis (TGA) was performed on the cured samples using a TG-92 Setaram thermal analyzer. The thermographs were obtained at a heating rate of 10°C/min using 10–15 g of the powdered sample. The experiments were made in a static air atmosphere.

Fire performance tests, including time to ignition (TTI), rate of heat release (RHR), time to reach maximum RHR, smoke density, carbon monoxide and carbon dioxide evolution, and sample mass loss, were determined by cone calorimeter in accordance with the procedure described in an ASTM standard method.<sup>14</sup> The heat flux produced was 50kW/m<sup>2</sup> on the specimen, which had an exposed surface of 100 × 100 mm. The testing equipment consisted of a radiant electric heater in trunk-conic shape, an exhaust gas system with oxygen monitoring and instrumentation to measure the gas flux, an electric spark for ignition, and a load cell to measure the weight loss. The test was terminated after 500 s of exposure. TTI measures the time to achieve sustained flaming combustion at a particular cone irradiance. Smoke density is measured by the decrease in transmitted light intensity of a helium-neon laser beam photometer, and expressed in terms of specific extinction area (SEA), with units of m<sup>2</sup>/kg. For RHR, the maximum value and the average to 180 s after ignition and the overall average values are determined. The total mass after the desired test time was calculated as a percentage of the initial sample mass.

## RESULTS AND DISCUSSION

### XRD analysis

Figure 1 shows the X-ray diffraction patterns of kaolin as received and the geopolymer-epoxy composites depending on the geopolymer synthesis (mixing with kaolin, potassium silicate, and potassium hydroxide) time (5, 15, and 30 min) followed by 15 min mixing with the epoxy matrix. From Figure 1, it is clear that the distinct peaks A, B, C, D, and E of kaolin can be observed at 12.0, 19.2–21.2, 24.0, 26.5, and 35.0–40.0 2-theta angle. However, no peak was observed at 12.0 degrees for kaolin (001) in geopolymer-epoxy composite mixes at different times between 5 min–30 min. The kaolin peak at position "B" is significantly lower in intensity in the 5 min mix synthesized geopolymer-epoxy composites, and gradually disappears when the geopolymer is synthesized for 15–30 min. Similarly, kaolin peaks at positions "C," "D," and "E" significantly decrease, finally disappearing with longer geopolymerization times. No crystalline peak was observed in geopolymer-epoxy composites after reaction for 30 min. This XRD pattern suggests that during the geopolymerization of kaolin, the kaolin crystalline structure converts to an amorphous inorganic polymer due to the formation of an Si-O-Al network. The

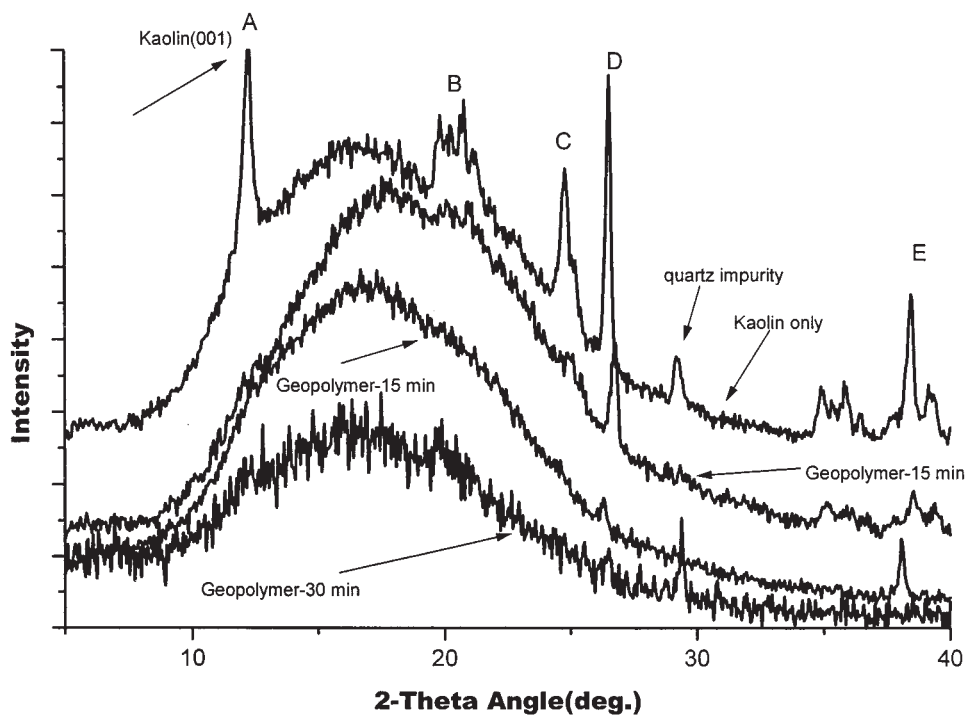


Figure 1 X-ray diffraction of kaolin and geopolymer-epoxy composites at various mixing times.

disappearance of kaolin characteristic peaks at A-E confirms a complete dissolution of crystalline kaolin particles during the geopolymerization. It is also confirmed from the XRD pattern that complete geopolymerization of kaolin required 15-30 min.

**FT-IR analysis**

The formation of geopolymers was further confirmed by FT-IR analysis. Figure 2 shows the FT-IR spectrum of DGEBA epoxy, kaolin, 20% geopolymer (5 min

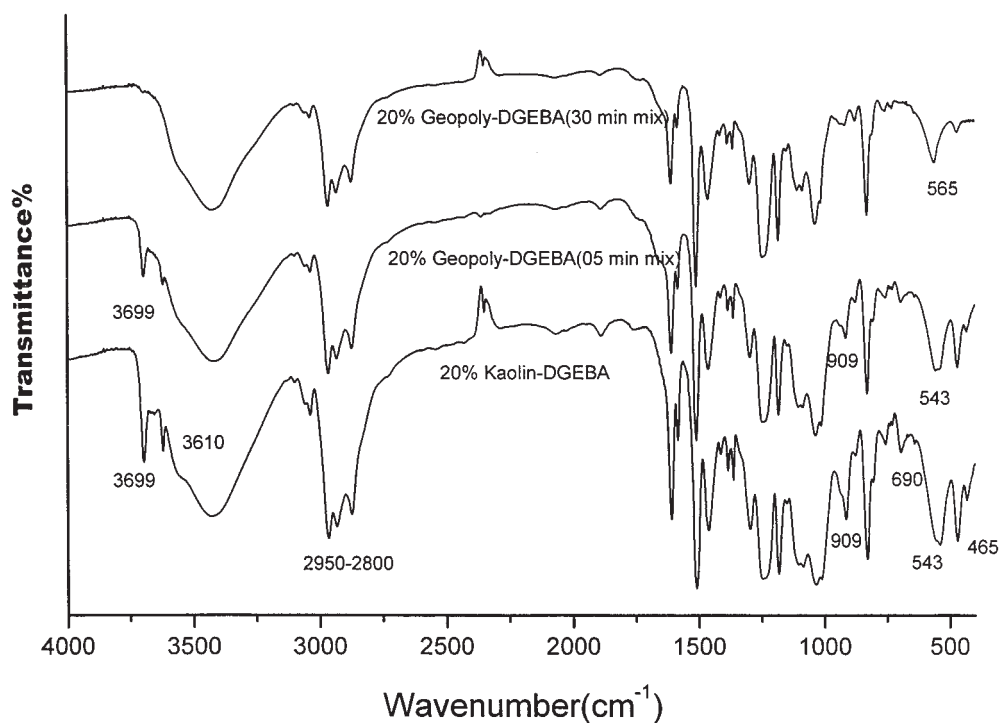


Figure 2 FT-IR spectra of kaolin-epoxy and geopolymer-epoxy composites.

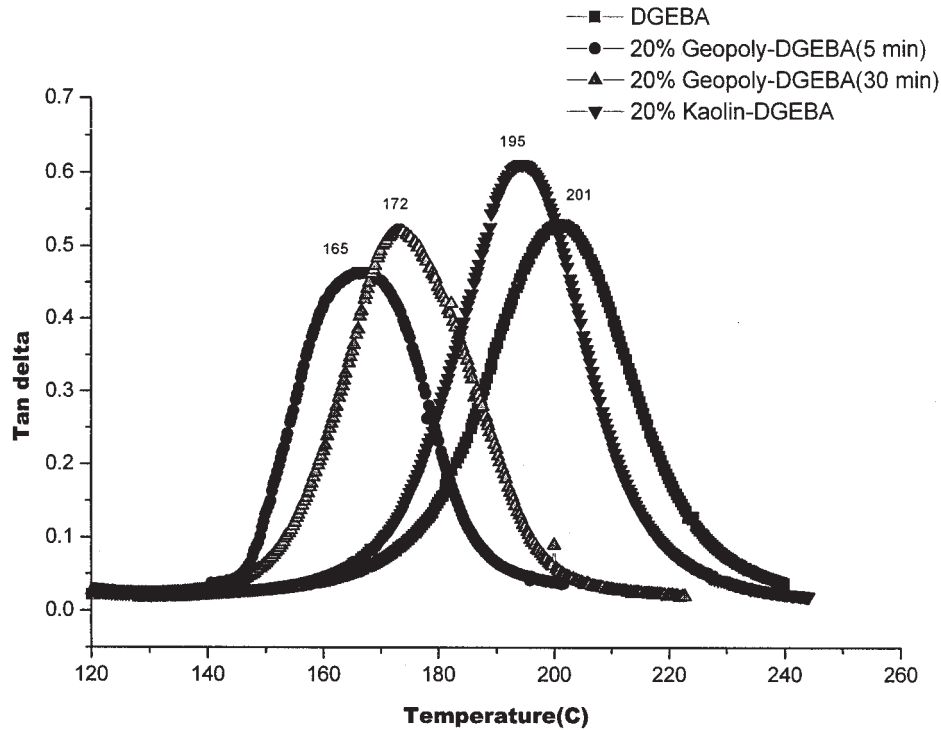


Figure 3 DMTA spectra of kaolin-epoxy and geopolymer-epoxy composites.

mixing), and 20% geopolymer (30 min) mixing. Characteristic kaolin stretching vibration  $\text{-OH}$  peaks were observed at  $3600$  and  $3610\text{ cm}^{-1}$ . However, these peaks gradually decreased with mixing time and fi-

nally disappear when mixed for 30 min due to geopolymerization. The broad peak assigned at  $3428\text{ cm}^{-1}$  is due to the presence of an  $\text{-OH}$  group in epoxy molecules. The phase transformation into geopolymers is

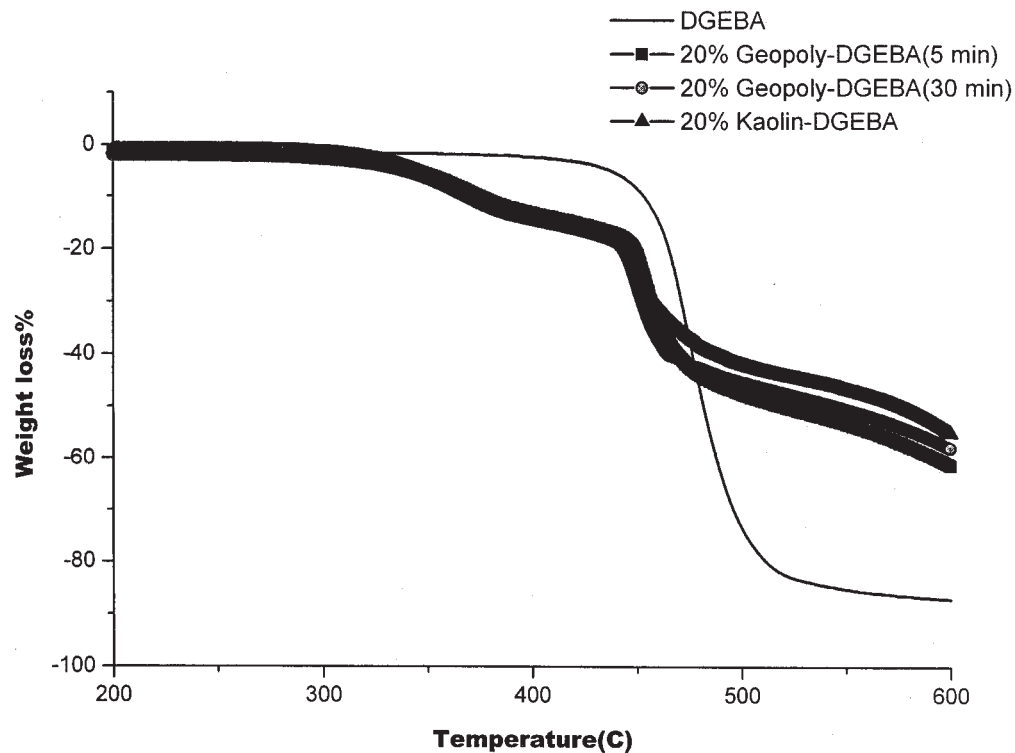


Figure 4 TGA spectra of kaolin-epoxy and geopolymer-epoxy composites.

**TABLE II**  
**Thermal Properties of DGEBA Epoxy and Modified DGEBA-Epoxy Composites**

	Temperature (°C) at 10% wt loss	Char yield 400°C	Char yield 450°C	Char yield 600°C
Pure DGEBA epoxy	463	100.0%	90.0%	16.0%
20% kaolin modified-DGEBA	384	90.0%	82.0%	46.5%
20% geo-poly modified-DGEBA (5 min mix)	382	89.0%	82.0%	42.0%
20% geo-poly modified-DGEBA (30 min mix)	380	88.0%	82.0%	39.0%

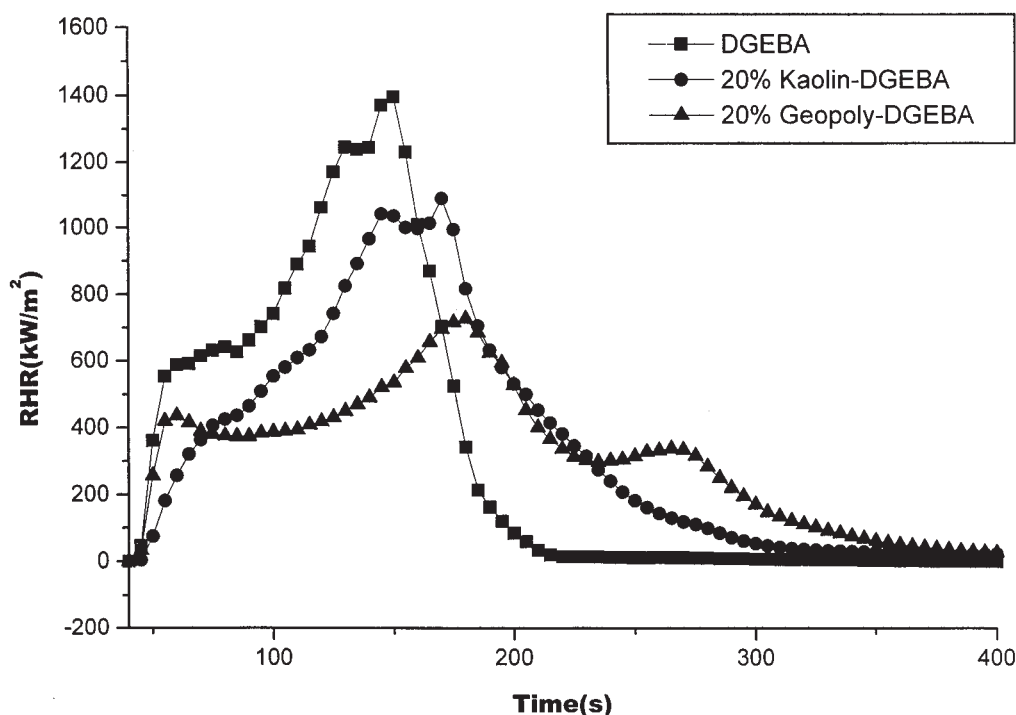
thus shown by the decrease and disappearance of various kaolin bands, namely the Si-O-Si symmetric stretching band at  $710\text{ cm}^{-1}$ .<sup>15</sup> Al-OH stretching in kaolin also decreased gradually with mixing time, and finally disappeared due to the formation of Al-O-Si bonds at  $910\text{ cm}^{-1}$ . The band at  $540\text{ cm}^{-1}$  is characteristic of the octahedral co-ordinate Al shifting to higher wavelength. The band at  $465\text{ cm}^{-1}$  in kaolin-DGEBA composites was found for kaolin bending Si-O-Si and O-Si-O. However, this gradually disappeared due to formation of the geopolymer zeolite phase.<sup>16</sup>

### Thermal properties of composites

#### Dynamic mechanical thermal analysis (DMTA)

The glass transition temperature and dynamic-mechanical properties of DGEBA, 20% kaolin-DGEBA, and 20% geopolymer-DGEBA were determined. Figure 3 shows the  $\tan \delta$  spectra from dynamic mechanical analyses of the cured DGEBA and its composites,

respectively. Cured DGEBA showed a  $T_g$  around  $201.0^\circ\text{C}$ ; addition of kaolin reduces this value to  $195^\circ\text{C}$ . The effect on the  $T_g$  of clay addition has been widely studied by many researchers, some reporting an increase in  $T_g$ ,<sup>17,18</sup> others finding a slight decrease or no change.<sup>19,20</sup> Becker et al.<sup>21</sup> reported that a decrease in  $T_g$  of clay-modified composites is due to interference of the clay with crosslink density, epoxy homopolymerization, and plasticization. The incorporation of geopolymers into the DGEBA epoxy showed an even lower  $T_g$  of around  $170^\circ\text{C}$ . The geopolymer-DGEBA composites prepared by mixing for 5 min showed an even lower  $T_g$  than those of composites prepared by mixing for 30 min. This confirms that complete geopolymerization is not able to occur when mixed for 5 min although it is complete after mixture for 30 min. The lower crosslink density of kaolin-modified DGEBA caused by the introduction of the filler is confirmed by the greater height of the  $\tan \delta$  peak, shown in Figure 3, which is indicative of greater relaxation strength. The lower relaxation strength and  $T_g$  of geopolymer-mod-



**Figure 5** RHR spectra of kaolin-epoxy and geopolymer-epoxy composites.

**TABLE III**  
Cone Calorimetric Data Measured With an Irradiance of  $50\text{kWm}^{-2}$

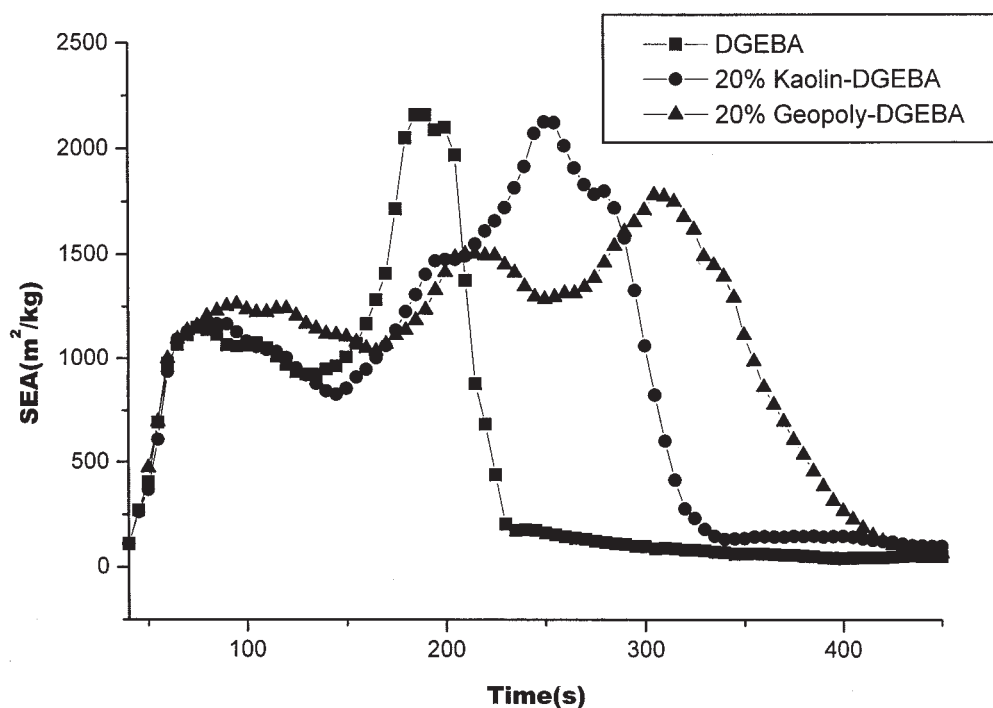
Fire properties	DGEBA	20% kaolin-DGEBA	20% geo-poly-DGEBA
Time to ignition(s)	65	69	60
Peak RHR ( $\text{kW/m}^2$ )	1396	1100	735
Av. HRR( $\text{kW/m}^2$ )	399.8	426	364.1
Av. HRR at 180s	501	562.5	471
Time to max HRR(s)	155	170	185
Av. effective heat of combustion ( $\text{MJ/kg}$ )	21.2	25.3	24.7
Av. CO yield ( $\text{Kg/Kg}$ )	0.0454	0.06	0.059
Av. $\text{CO}_2$ yield ( $\text{Kg/Kg}$ )	1.48	1.99	1.955
Total heat evolved ( $\text{MJ/kg}$ )	89.65	115.24	95.68
Mass loss %	82.8	73.9	74.7

ified composites are believed to be due to structural inhomogeneity, later explained by SEM images.

#### Thermogravimetric analysis (TGA)

Thermogravimetric analysis was performed to examine the effect of geopolimer and kaolin addition on the thermal stability of cured DGEBA. Figure 4 shows the weight loss for the unmodified DGEBA epoxy, kaolin modified DGEBA, and different geopolimer-DGEBA composites. Pure DGEBA epoxy resin shows a one-step degradation mechanism. However, kaolin or geopolimer modified DGEBA showed a three-step degradation mechanism. Table II reflects the thermal properties of pure DGEBA epoxy resin and modified-DEGBA epoxy resin. In

the unmodified, cured DGEBA system, initial degradation commences at around  $430^\circ\text{C}$ ; however, the rate of degradation significantly increases above  $450^\circ\text{C}$  and results in a char yield of 16% remaining at  $600^\circ\text{C}$ . The degradation temperature at 10% weight loss was  $463^\circ\text{C}$  for DGEBA and around  $380\text{--}384^\circ\text{C}$  for modified DGEBA composites. These results indicate that the thermal stability of the modified epoxy resin at lower temperature ( $<400^\circ\text{C}$ ) is found not superior to that of the pure DGEBA epoxy resin. However, the char yield of the modified epoxy resin at  $600^\circ\text{C}$  is much higher than that of the unmodified DGEBA epoxy resin. High char yield formation prevents the production of combustible gases material and thus decreases the thermal conductivity of the surface of the burning materials.<sup>22</sup>



**Figure 6** SEA spectra of kaolin-epoxy and geopolimer-epoxy composites.

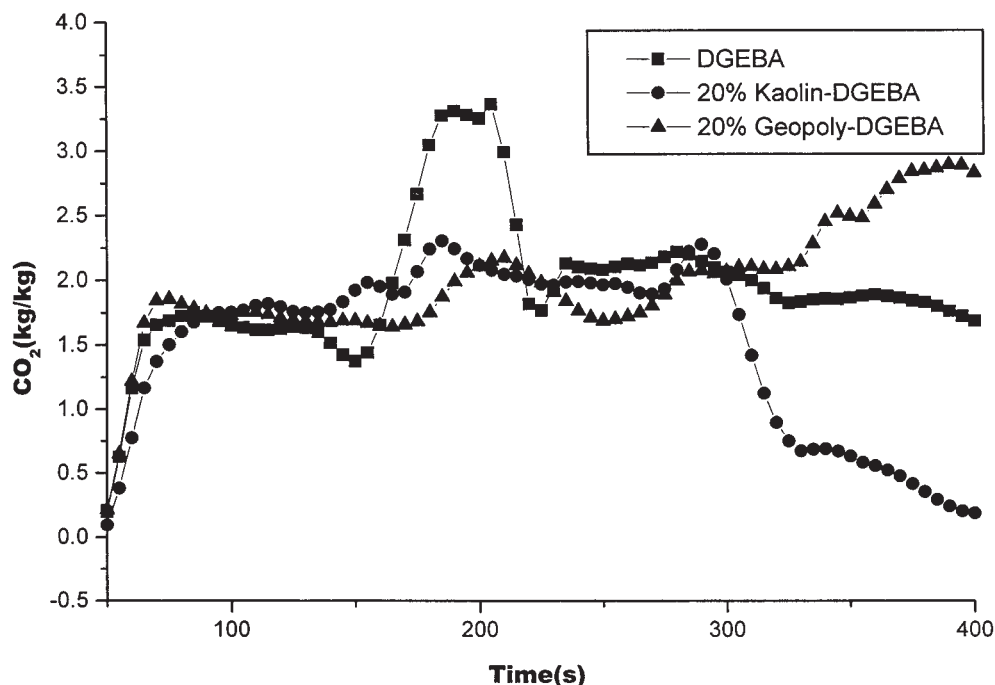


Figure 7 CO<sub>2</sub> emission of spectra of kaolin-epoxy and geopolymer-epoxy composites.

Flammability testing by cone calorimetry

The cone calorimeter provides important information on the combustion behavior of a material under ventilated conditions. The peak rate of heat release of a material is one of the important factors to determine the potential behavior during fire. Figure 5 shows the rate of heat release (RHR) of unmodified DGEBA and

modified DGEBA variation with time at a heat flux of 50 kWm<sup>-2</sup>. The peak rate of heat release for DGEBA is high at around 1400 kWm<sup>-2</sup> after 150 s. However, 20% kaolin modified DGEBA has a lower peak rate of release at 1100 kWm<sup>-2</sup>, significantly reduced by 21.5% compared to unmodified DGEBA. In contrast, DGEBA modified with 20% geopolymer showed a peak release

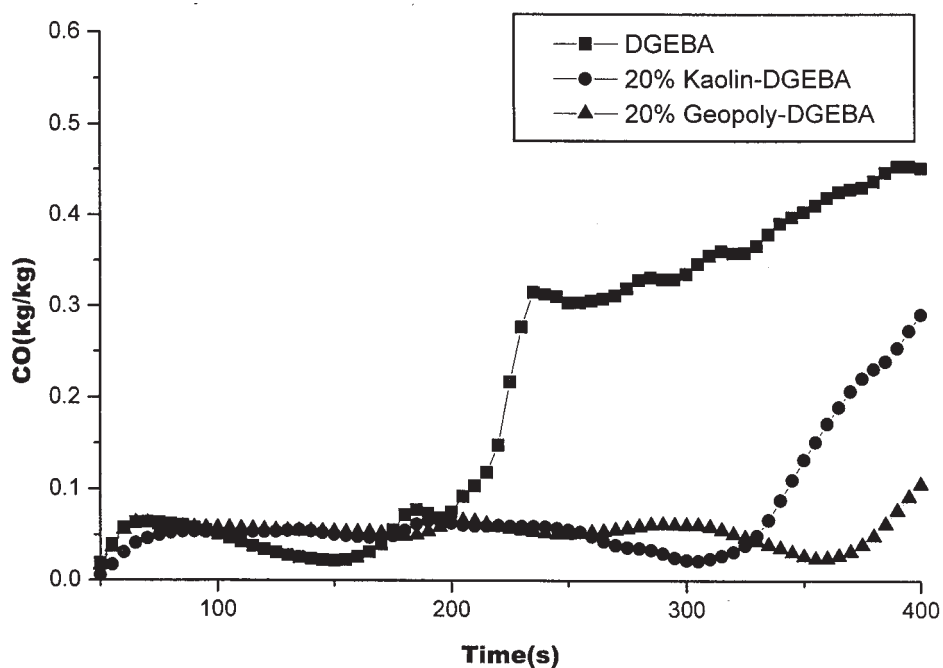


Figure 8 CO emission spectra of kaolin-epoxy and geopolymer-epoxy composites.



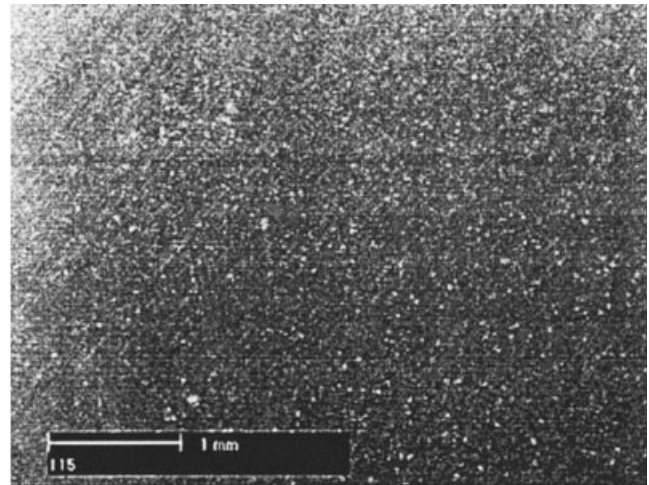
**TABLE IV**  
**Fire Performance Index of Unmodified DGEBA and Modified DGEBA System**

DGEBA system	Peak RHR (kW/m <sup>2</sup> )	Time to ignition (s)	FPI (sm <sup>2</sup> /kW)
DGEBA only	1396	65	0.046
20% kaolin-DGEBA	1100	69	0.062
20% geopolymer-DGEBA	735	60	0.081

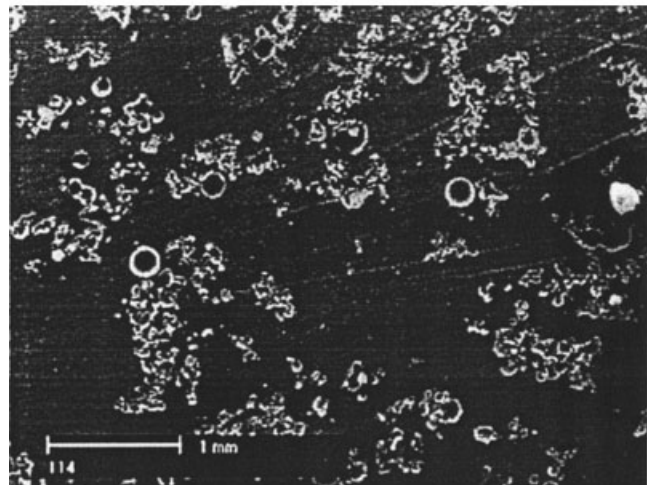
rate at 702 kWm<sup>-2</sup>, which is 47% lower than that of unmodified DGEBA. An increase in flame retardancy in modified DGEBA can be attributed to the residual masses or char obtained during firing. A higher percentage of mass residue or char indicates a condensed-phase flame retardance mechanism.<sup>23</sup> Other important parameters obtained from the epoxy and modified epoxies by cone calorimetry are given in Table III.

Evaluation of the fire performance of epoxy resins involves quantifying smoke generation at specific extinction areas (SEA) and quantifies production of CO and CO<sub>2</sub>. SEA, which measures the total obscuration area of smoke produced, divided by the total mass loss during burn. Figure 6 shows the SEA of DGEBA and kaolin/geopolymer modified DGEBA as a function of time. The SEA value of DGEBA was found to be maximum around 200s and decreased rapidly. However, modified DGEBA showed a relatively lower value at 200s and maximum value observed at 250 s and 325 s for kaolin and geopolymer modified DGEBA, respectively. This can be explained by the fact that unmodified DGEBA readily converts into smoke more easily when it burns and does so over a longer period of time. CO<sub>2</sub> emission during burning shows in Figure 7 that the CO<sub>2</sub> emission rate is always higher for the unmodified DGEBA system compared to the modified epoxy resin system. The maximum CO<sub>2</sub> emission was observed after 200s of ignition. The lower production level of CO at 200s of modified and unmodified DGEBA is shown in Figure 8. However, CO level drastically increases for unmodified DGEBA resins after 200s and reaches maximum CO production of 0.45 kg/kg with respect to time. CO emission for the modified DGEBA system remained constant for a longer period of time. The higher CO production of unmodified DGEBA indicates incomplete combustion, and possible for the gas phase activity. Thus, the addition of kaolin or geopolymer showed the reduction of RHR.

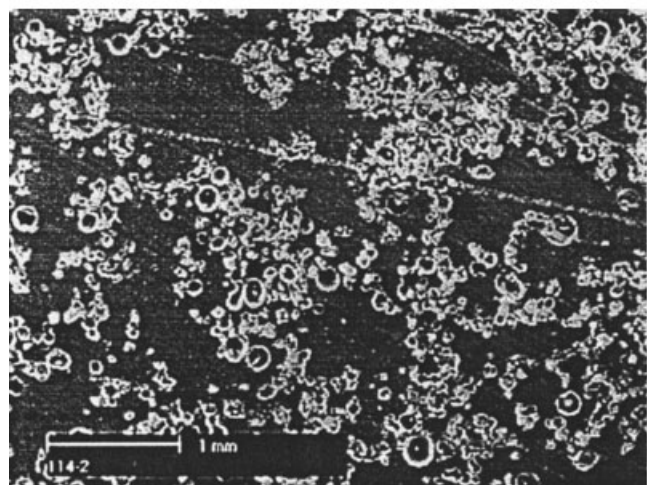
Fire performance of a material can also be calculated from the fire performance index (FPI), which is the ratio between the time of ignition (time) and the peak rate of heat release (RHR). Table IV shows the fire performance index of the unmodified-DGEBA and the modified DGEBA system. DGEBA, without any modification, shows the lowest fire performance at 0.046 sm<sup>2</sup>/kW. However, when DGEBA is modified with



(a) 20% Kaolin-DGEBA



(b) 20% Geopolymer-DGEBA(5 min mixing)



(c) 20% Geopolymer-DGEBA(30min mixing)

**Figure 9** SEM images of spectra of kaolin-epoxy and geopolymer-epoxy composites.

kaolin, the FPI increased to 0.062, an increase of 35%. In comparison, the geopolymer modified DGEBA FPI increased to 0.081  $\text{sm}^2/\text{kW}$  and improved 76%.

### Microscopic morphology

Figure 9 shows the SEM micrographs of 20% kaolin modified-DGEBA and 20% geopolymer-DGEBA mixed for 5 min and 30 min, respectively. It is clear from Figure 9(a) that kaolin particles were homogeneously dispersed into the DGEBA matrix. No agglomeration was observed. However, in the geopolymer-DGEBA system, a different microstructure was observed. When the geopolymer was prepared by 5 min mixing, a discontinuous island like structure was observed, as shown in Figure 9(b). This discontinuous island like structure gradually increased to a cocontinuous structure when the geopolymer was prepared by 30 min mixing, as observed in Figure 9(c). It is evident from the micrographs that incorporation of the geopolymer into the organic polymeric system is possible with good microstructure. This type of microstructure, however, showed a lower relaxation behavior and  $T_g$  but exhibited higher thermal stability and higher fire performance capability. Other properties of materials, mainly the mechanical properties, before and after firing tests of the composites should be investigated further.

### CONCLUSIONS

In this experiment, inorganic polymers (geopolymers) were incorporated into an epoxy resin system to investigate the fire performance capability. The addition of only 20% kaolin into the DGEBA epoxy matrix shows the thermal stability was improved. This improvement was further increased when 20% geopolymer was added into the epoxy system. The probability of chemical bonding between the inorganic polymer and the organic polymer is also under investigation.

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