



The mechanical and thermal characteristics of phenolic foams reinforced with carbon nanoparticles



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ABSTRACT

Recently phenolic foams have been spotlighted because of their excellent flame resistance, high thermal stability over a broad temperature range and low generation of toxic gases during combustion. However, phenolic foams have inferior mechanical strength and high thermal conductivity compared with other polymer foams, and it is difficult to control the cell morphology by forming different sizes of pores during the foaming and curing process because phenolic resins contain various solvents. In this work, the nanoparticles such as multi-wall carbon nanotubes (MWCNTs) and graphene-reinforced phenolic foams were fabricated to control the cell morphology. To improve the thermal and mechanical properties of the phenolic foam, the proper foaming point of each particle-reinforced phenolic foam was investigated by cure monitoring using a dielectric sensor. From the experimental results, the effects of viscosity and the dispersion of particles on the cell morphology was introduced, and the optimal weight fraction of each particle was found based on the cell morphology and the thermal and mechanical properties of the phenolic foams.

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1. Introduction

Polymer foams such as polyurethane and polystyrene foam are fabricated by using foaming agents, flame retardants, cross-linking agents and surfactants. However, the foaming agents such as CFCs and HCFCs have been restricted because they can cause ozone depletion and global warming [1]. Furthermore, the commercial polymer foams generate toxic gases during combustion so alternate materials and preparation methods have been required [1–3].

Phenolic foam has excellent flame resistance and low generation of toxic gases during combustion. It also has a high self-ignition temperature of 480 °C and high thermal stability over a broad temperature range, maintaining performance and stability from –196 °C to 200 °C. This has led to a broad range of applications as an insulating material [4,5]. However, phenolic foam has a high thermal conductivity compared with other polymers because of its high thermal conductivity (0.4 W/m K) in the solid state. Additionally, it is difficult to control the cell morphology by formation of various sizes of pore during the foaming and curing process because phenolic resins contain various solvents [5–7]. For this reason, the physical properties of conventional phenolic foam have not been able to satisfy the insulation specifications for

structural members. The physical performance of different polymer foams was determined by their cell density and cell uniformity [8–10]. The cell density of the foam is affected by the degree of cure and the viscosity before the foaming, the type of reinforcing particle, and the weight fraction of the particles.

Many studies have focused on the improvement of the physical properties of polymer foams. Nemoto et al. prepared the nano-micro cellular foam from a polypropylene/propylene-ethylene copolymer blend by controlling the viscoelasticity and CO₂ solubility [11]. Song et al. studied the effect of viscosity on aluminum structure, pore distribution and pore diameter [12]. However, there is a certain limit to improving the material properties by changing process parameters. In other studies, different types of organic and inorganic fillers were used to improve the strength and stiffness of polymer foams [13–18]. Desai et al. had shown that the mechanical properties of phenolic foams were improved by adding a 1:1 ratio of glass to aramid fiber [13]. Zhang et al. found that the strength of the polymer foams with multi wall carbon nanotube (MWCNT) and functionalized MWCNT were increased by 73% and 74%, respectively [14]. Chen et al. increased the mechanical properties of the closed-cell type PMMA foam by adding the MWCNT [15]. In addition, many studies investigated the effect of nanofillers on the improvement of mechanical properties by preventing crack propagation and increasing the interaction between the matrix and the particles [16–20]. Yang et al. fabricated the phenolic foam with

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functionalized multiwalled carbon nanotubes to improve the mechanical properties and fire resistance [21]. As introduced above, most studies on the particle-reinforced polymer foams explained the effects of reinforcing particles on mechanical properties, but analyses of the effect of the particles on cell formation by influencing the resin viscosity and cross-linking of the resin are rare.

Attaining optimal viscosity before the foaming process increased the cell density because the viscosity of the resin could control the expansion of the pores during the foaming process [12]. The initial degree of cross-linking in the resin influences the viscosity of the resin. As cross-linking begins, the viscosity increases rapidly and prevents the expansion of the pores during foaming. Conversely, when the polymer foam was formed after the cross-linking has already progressed, cell formation was interrupted by the resulting high viscosity. Therefore, when particles were used to reinforce the polymer resin, a determination of the optimal foaming point is important to obtain the required mechanical and thermal properties of the polymer foam [22].

In this work, the phenolic foams were reinforced with two types of carbon nanoparticles, multi-wall carbon nanotube and graphene. They were fabricated to improve the mechanical and thermal performance by a short-period microwave foaming process. To investigate the effect of the particles on the cross-linking and viscosity of the phenolic resin, cure monitoring was performed by using a dielectric sensor and DSC analysis. Based on these results, the optimal foaming point was determined to obtain optimal cell morphology and cell density. The cell morphology was estimated by foam density and porosity, and confirmed by scanning electron microscope (SEM) images. Mechanical and thermal properties were characterized by compressive tests and thermal conductivity tests using varying weight percents of different particles. To evaluate the thermal stability as temperature increases, weight loss was measured by using TGA. Finally the optimal foaming condition for each of the particle-reinforced phenolic foams was suggested based on the measured mechanical and thermal properties.

2. Materials and experimental procedures

2.1. Materials

Resole-type phenolic resin (OG-5000, Kangnam Chemical Co., Korea) was mixed with an acid catalyst (PTSA 65%, Kangnam Chemical Co., Korea) in a mixing ratio by weight of 9:1. The resin and catalyst were kept at 15 °C to control the viscosity change before mixing. The mixture was then stirred at 500 rpm for 1 min using the impeller. Multi-wall carbon nanotube (724769, Sigma-Aldrich Co. LLC., USA) and graphene (C500, XG Sciences Inc., USA) were added to the phenolic resin at different weight

fractions of 0.5, 1.0 and 2.0 wt%. Table 1 shows the physical properties of the phenolic resin and the reinforcing particles.

2.2. Preparation procedures for the phenolic foam

In our previous work, a study of the mechanical and thermal properties of the phenolic foam was performed with respect to the aging time before microwave foaming. To determine the optimal foaming point, the degree of cure of the phenolic resin was monitored in real time. As a result, the phenolic foam which was fabricated by applying the microwaves at the cure starting point showed highly improved thermal and mechanical characteristics [22].

Based on these previous results, cure monitoring of the carbon nanoparticle-mixed phenolic resin was performed to decide the cure starting point with respect to the particle type and weight fraction (wt%) at room temperature (25 °C). MWCNT and graphene were mixed with the phenolic resin at different weight fraction of 0.5, 1.0 and 2.0 wt%, respectively, and their effect on the cure reaction of the phenolic resin was monitored. Each sample was named with respect to the particle type and weight fraction as shown in Table 2. To estimate the qualitative degree of cure of the mixture as a function of the aging time after mixing the phenolic resin and cure accelerator using an impeller, a dielectric sensor (Lacomtech, Republic of Korea) and a K-type thermocouple (TT-K-30, OMEGA, USA) were used to measure the dissipation factor and the temperature, respectively. The dissipation factor D was measured in the phenolic resins using a commercial dielectrometer (ELC-133A, Escort Instruments Corp., USA) using a 1 kHz alternating current. Each sample was tested in triplicate to determine the repeatability of the results.

The isothermal DSC, (Q20, TA Instruments, USA) experiments were carried out at room temperature to compare cure monitoring results by using the dielectric sensor. All of the uncured samples were ramped from 15 °C to 25 °C with a heating rate of 5 °C/min and were kept isothermal for 50 min. This procedure is similar to the cure monitoring condition using the dielectric sensor. All DSC tests used aluminum sample pans and 50 mL/min of N₂ purge.

The resin viscosity was measured with respect to particle type and weight fraction using a viscometer (RVDV-II + P, Brookfield, USA). The viscometer is a rotational apparatus which measures the torque required to rotate a spindle at constant speed immersed in the resin of the given temperature. The average resin viscosity was calculated from the torque data measured for 5 min at 25 °C.

Based on the cure monitoring results, the microwave foaming was performed using the following three steps as shown in Fig. 1: (1) mixing the resole and accelerators with or without the particles using an impeller at 500 rpm for 3 min; (2) aging the mixture under constant room temperature to control the initial degree of cure before microwave foaming; and (3) foaming by microwave using an effective intensity per unit mass of 12 kW/kg at 2.4 GHz and de-molding. [22].

Table 1
Physical properties of the neat phenolic resin and the carbon nanoparticles.

	Phenolic resin (OG-5000)	
Viscosity (Pa s)	13–15 (25 °C)	
pH	6.5	
Density (g/cm ³)	1.13	
	MWCNT	Graphene
Carbon content	>95%	>98%
Bulk density	~2.1 g/mL	0.2–0.4 g/mL
Dimensions	6–9 nm × 5 μm (O.D. × L)	500 m ² /g, <2 nm (BET, thickness)

Table 2

Cure starting point as a result of dielectric cure monitoring and the time at the maximum exothermic peak for isothermal scanning at room temperature (25 °C) of the phenolic resins.

Sample	Particle content	Cure starting point (min)	Max peak (min)
NP	–	17.74 ± 0.69	2.14
CP 0.5	0.5 wt% MWCNT	16.33 ± 0.50	1.84
CP 1.0	1.0 wt% MWCNT	15.58 ± 0.30	1.82
CP 2.0	2.0 wt% MWCNT	15.91 ± 0.33	1.99
GP 0.5	0.5 wt% Graphene	17.59 ± 1.39	1.93
GP 1.0	1.0 wt% Graphene	17.37 ± 0.70	1.76
GP 2.0	2.0 wt% Graphene	17.51 ± 0.92	1.83

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