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Novel foaming methods to fabricate activated carbon reinforced microcellular phenolic foams

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ABSTRACT

Polymer foams are used for thermal insulation and weight reduction in multiple fields, such as buildings, automobiles, and LNG containment systems. Phenolic foams are preferred as a thermal insulator due to a lower flammability and lower gas generation than other polymeric insulation foams. For conventional foaming methods, the extensive time to fabricate large volumes and environmental regulations limit the use of blowing agents. In this study, new foaming methods for an activated carbon (AC) reinforced microcellular phenolic foam were developed using microwaves instead of the blowing agents. Both thermal and mechanical properties were measured to characterize the foaming conditions, including the aging time of the resin before microwave foaming and the weight percent of the AC. We found that the microcellular phenolic foams fabricated under these optimum foaming conditions had low thermal conductivities and low densities that are suitable for insulating foams.

1. Introduction

Commercial insulation foams such as polystyrene and urethane foams are widely used in sandwich structures as insulation due to their low density and low thermal conductivity [1–3]. These characteristics are changed by cell morphology. The foam consists of a polymer matrix permeated with either air bubbles or air tunnels, designated as either a closed cell or an open cell structure, respectively. Closed cell foams are generally more rigid, while open cell foams are usually flexible [4–7].

Polystyrene and urethane foams have closed cell or open cell structures, but they have lower thermal stability and also produce toxic gases during combustion, causing environmental concerns. For this reason, phenolic foams are of interest due to a low flammability, low generation of toxic gases during combustion, excellent resistance to flame and a high self-ignition-temperature of 480 °C [8,9].

Furthermore, the entire foam industry has been challenged in recent years by issues ranging from recyclability to the depletion of the Earth's ozone layer by chlorofluorocarbon blowing agents such as CCl_3F. These challenges have been further compounded by governmental regulations [10]. As a result, it has become necessary to develop new approaches using either environment-friendly blowing agents or no blowing agents at all.

Microwave foaming technology is a simple and cost effective method. The polar group vibration by microwaves can form phenolic foams without blowing agents in a short period of time. After mixing the resole type phenolic resin with the cure accelerators, a H_2O by-product is produced during the cure process as shown in Fig. 1a. The resole can be cured at 150–250 °C without a cure accelerator, although the long time needed to complete the cure reaction gives the air bubbles a chance to escape from the resin. The temperature inside of the resole must be increased to the cure temperature quickly and uniformly to expand the air bubbles. This process can be accomplished with microwave heating, because the microwave vibrates all of the molecules with a polarity similar to the resole and the H_2O generated from the cure reaction. The presence of H_2O in the resin can increase the exited state, causing an increase in the cell size during the microwave heating process. To ensure a low thermal conductivity of the phenolic foams, it is necessary to control the H_2O generated during the cure reaction.

In this work, The AC reinforced microcellular phenolic foams was fabricated to improve the mechanical and thermal properties of the foams by microwave method in a short time. The very large surface area of the porous AC allows for an adsorption by chemical interactions with the phenolic resin in curing reaction [11].

The mechanical properties and the cell structures of the phenolic foam were characterized based on the aging time of the phenolic resin before the microwave foaming process. The aging time can affect the morphologies and thermal and mechanical properties of the phenolic foam because of the viscosity changes by curing reaction. The cure monitoring was performed by a dielectric sensor to

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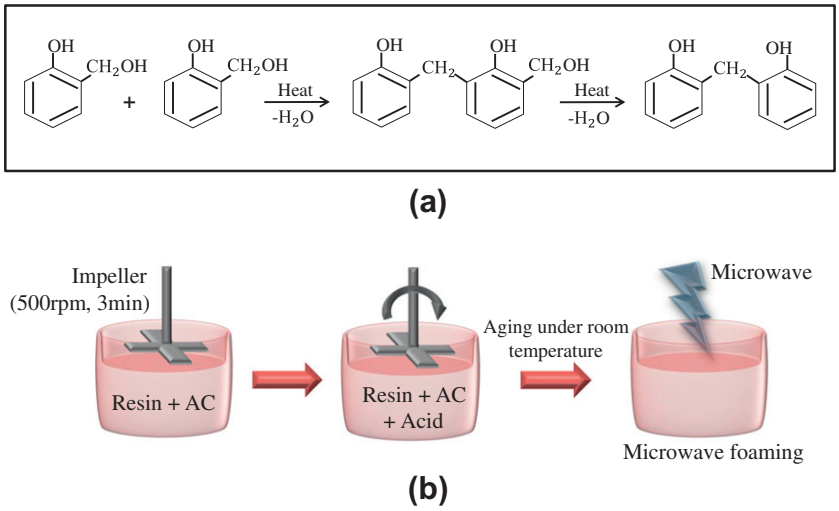

Fig. 1. Schematic diagram of the microwave foaming process and the cure reaction of a resole type phenolic resin: (a) chemical cure reaction, (b) procedures for the microwave foaming.

decide the optimum aging time before microwave foaming. The mechanical properties of the phenolic foams characterized by compressive test at different aging time and thermal properties were estimated by hot wire-method and TGA.

2. Materials and testing methods

2.1. Materials

Two resole-type phenolic resins, OG-5000 (Kangnam Chemical Co., Korea) and CA666 (Kangnam Chemical Co., Korea), were mixed with two types of cure accelerators, PTSA 65% (Kangnam Chemical Co., Korea) and nitric acid (DAE JUNG, Korea). Table 1 shows the physical properties of the two resole type phenolic resins. The size distribution of the AC powder (325 mesh, Kaya AC Inc., Korea) used for the purpose of reinforcements and gas adsorption was 10–350 μm.

2.2. Microwave foaming

To select an optimum combination of the resin and cure accelerators, preliminary microwave foaming experiments were performed with the materials shown in Table 2.

The microwave foaming was performed using the following three steps: (1) mixing the resole and accelerators with or without the AC 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 as shown in Fig. 1b [12].

The AC was used to control the cell size by trapping the generated gas such as H_2O during the curing reaction and to reinforce the foam structure. The optimum weight percent of the AC was determined from the foam morphology of various samples. At least five specimens were tested for each sample type to determine the repeatability of the results. Based on the preliminary test results, the addition of the AC and the aging time before microwave foaming to control the initial degree of cure were selected as test parameters as shown in Table 3.

Table 1
Properties of resole type phenolic resins.

Sample	OG-5000	CA-666
Viscosity (cps)	4000–7000 (25 °C)	1000–2000 (25 °C)
pH	6.5–7.5	8.5–9.2
Density (g/ml)	1.132	1.15

Table 2
Combinations of the resins and cure accelerators.

Sample	Resin:acid
CA:nitric acid	90:10
CA:PTSA	90:10
OG:nitric acid	90:10
OG:PTSA	90:10

2.3. Characterization

2.3.1. Cure monitoring of the phenolic resins

To estimate the degree of cure of the phenolic resins in accordance with the aging time after mixing the phenolic resins and the accelerators using an impeller, a dielectric sensor (Lacomtech, South Korea) and a K-type thermocouple (TT-K-30, OMEGA, USA) were used to measure the dissipation factor and the temperature (approximately room temperature), respectively. For the dielectric sensor measurements, two electrodes embedded in the phenolic resin were connected to an alternating electric field, forming a capacitor with the phenolic resin. The charge accumulated in the capacitor is dependent on the mobility of dipoles and ions, which are present in the resin and vary with cure state. The degree of cure is related to the movement of the dipoles and ions, which have a high mobility when the phenolic resin is uncured. Their movement is restricted abruptly when the phenolic resin enters into a gel state or solidifies. The degree of movement can be expressed by

Table 3
Experimental parameters to control the cell morphology and performance.

Sample	Resin:acid:AC wt%	Aging time
SP1	90:10	3.7 min
SP2	90:10	4.4 min
SP3	90:10	5.1 min
SP4	90:10:1 wt%	3.7 min
SP5	90: 10:1 wt%	4.4 min
SP6	90: 10:1 wt%	5.1 min