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# A microwave foaming method for fabricating glass fiber reinforced phenolic foam



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#### ABSTRACT

Phenolic foam is a fire-retarding insulator that can endure temperatures of up to 300 °C, and it is carbonized when it is exposed to fire, which decreases its thermal conductivity and prevents the spread of fire. Therefore, phenolic foam has been used for many applications, such as in the architectural and aerospace fields. However, phenolic foam requires blowing agents during the fabrication process, which causes environmental pollution. Therefore, a new foaming method that is environmentally friendly is needed. In addition, due to the brittleness of phenol, a fiber reinforcement method is required to improve the mechanical properties of the phenolic foam.

In this study, a new foaming method for fabricating low-density phenolic foam that uses microwaves is developed and optimized. The effect of the viscosity of the phenolic resin and the curing speed on the foam density and uniformity is investigated. To improve the mechanical properties of the phenolic foam, a reinforcement method using chopped glass fiber was optimized with respect to the fiber length and content. The mechanical properties of the fiber reinforced foam were evaluated by measuring the tensile strengths and fracture toughnesses. Finally, the thermal conductivity of the phenolic foam fabricated by the microwave foaming method is measured.

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#### 1. Introduction

Phenolic resin has been used in various industries, such as aerospace and construction, and in military applications because it has fire retarding characteristics. The decomposition of phenolic resin starts at 575 K, and the smoke density is low compared with other polymers [1,2]. In addition, only harmless carbon dioxides and water are generated when phenol burns, moreover char is formed by carbonization, which is mechanically stable even at high temperature and prevents the spread of fire [3–5]. Therefore, the phenolic resin has been introduced for fire protection purposes.

Polymeric foams are typical insulators and generally used in buildings, refrigeration systems, and in LNG containment [6–9]. In the past, polystyrene and polyurethane foams were commonly used [10,11], but the use of these foams has been decreased due to environmental and fire safety issues because they generate toxic combustion gases, such as hydrogen cyanide and styrene oxide [12]. In contrast, phenolic foams generate much less smoke, which mainly consists of carbon dioxides and water [13]. In addition, if the phenolic foam is carbonized by fire, the phenol is converted

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http://dx.doi.org/10.1016/j.compstruct.2016.05.044 0263-8223/© 2016 Elsevier Ltd. All rights reserved. to char, which can provide a structural support at high temperature [1]. Therefore, conventional foams are being substituted by phenolic foams, and there are many commercial phenolic foams on the market these days.

In fact, the fabrication method for phenolic foams requires a blowing agent such as chlorofluorocarbons (CFCs) or hydrochlorofluorocarbons (HCFCs), which cause global warming because they deplete the ozone layer, increasing the exposure to ultraviolet rays from the sun [7]. Hence, many countries have been trying to reduce the use of these since 1987 following the establishment of Montreal Protocol [14]. However, the conventional method of fabricating phenolic foams is not easy to avoid the use of such blowing agents, which also increases the production cost.

Basically, the phenolic resin contains some alcohol as a solvent, and the curing reaction generates water as a by-product. A cellular structure can be formed if the solidification of phenolic resin occurs simultaneously during the curing process. In addition, if micro air bubbles are embedded in the resin, the cured phenol may become more porous. However, in the past, there was no proper method to uniformly heat the resin. In the case of typical heating using an oven, the outer resin is cured first where the heat is transferred, which prevents escaping of moisture that makes the quality of the foam very poor. Therefore, the foaming of commercial foams







is performed at room temperature using blowing agents such as CFCs or HCFCs.

Since the phenolic resin is a polar molecule, microwaves are applicable for its heating. The electromagnetic microwaves can increase the temperature of the resin uniformly and quickly, making it possible for a uniform foam structure to be obtained. Furthermore, if the temperature reaches higher than 100 °C, the water produced as a by-product and the alcoholic solvent may evaporate to escape from the foam. Then air will substitute these gases, and it will decrease the thermal conductivity of the foam because air has much lower thermal conductivity than water or water vapor. Therefore, microwave foaming may be a feasible method for manufacturing phenolic foam.

In a previous study, Kim et al. suggested a microwave foaming method for phenolic foam that enables the fabrication of high density foams of approximately 100 kg/m<sup>3</sup>, and the feasibility of the microwave foaming was verified [15,16]. However, the typical density of phenolic foam used as an insulator for the lowest thermal conductivity is between 30 and 40 kg/m<sup>3</sup> [17], and foam with a lower density and thermal conductivity is preferred in actual applications. Therefore, it is necessary to decrease the density of the foam fabricated by the microwave foaming method, and the fabrication process should be optimized.

The other problem is the brittleness of phenolic foams, which leads to a possibility of damages during handling of phenolic foam [3]. The workability of building materials is important on the construction site, and the conventional phenolic foam could not achieve enough workability due to easy fracture. To improve the ductility of the phenolic foam, the fiber reinforcement should be adopted and optimized.

In this study, the microwave foaming method was used to fabricate low density phenolic foam. The effect of the resin viscosity and the curing speed of the phenolic resin on the foam density and uniformity was investigated, and the closed mold foaming was attempted. The resin viscosity was controlled by the amount of ethanol, and the curing speed was regulated by the amount of acid catalyst, which was a 65% aqueous solution of p-toluenesulfonic acid (PTSA). In addition, the phenolic foam fabricated by the microwave foaming method was reinforced with chopped glass fiber to improve the fracture toughness. The tensile strengths and fracture toughnesses of the reinforced phenolic foam were measured with respect to the length and amount of chopped glass fiber. Finally, the thermal conductivity of the low-density phenolic foam specimen was measured and evaluated.

#### 2. Experimental

#### 2.1. Microwave foaming method

Fig. 1 shows a schematic diagram of the process of the microwave foaming method. The process consists of four steps: (1) preparing a resole-type phenolic resin, OG-5000 (Kangnam

Chemical, Co., Korea), in a mold; (2) entrapping micro air bubbles in the resin by impeller mixing at 3000 rpm for 60 s; (3) adding an acid catalyst (i.e., PTSA) and mixing again at 3000 rpm for 20 s; (4) foaming by microwave with an intensity of 1000 W at 2.4 GHz, and post-curing at 80 °C for 2 h. In step (1), ethanol was added to the phenolic resin, which decreases its viscosity, and the amount of PTSA was varied to control the curing speed.

### 2.2. Density and uniformity of the phenolic foam with respect to the resin viscosity and curing speed

To investigate the effect of the resin viscosity on the foam density, the free rise density of the foam using various phenolic resins with different viscosities was measured and compared. The resin viscosity was controlled by the content of ethanol from 0 to 20 wt. %. A dial viscometer (LVT Viscometer, Brookfield Engineering, USA) was used to measure the viscosity of the resin mixture. The viscosity was measured at 25 °C based on the ASTM standard [18]. The content of the PTSA catalyst was also varied, and 3 wt. % and 6 wt. % were adopted. The resin mixture of 2 g was foamed freely by the microwave foaming method. The masses were measured before and after foaming, and the average foam density was calculated.

In addition, closed mold foaming was attempted. The shape of the mold was cuboidal, with dimensions of  $80 \times 80 \times 100 \text{ mm}^3$  (= width × length × height). For resins with different viscosities, the resin mixtures of 50–60 g were also foamed. The exact mass was determined experimentally to obtain the average foam density of 35 kg/m<sup>3</sup>. The foaming process was the same as in Fig. 1. The air inside the mold was eliminated through the venting slit on the top of the mold. After the foam was demolded after post-curing, the morphology was observed and compared.

#### 2.3. Chopped glass fiber reinforcement

The phenolic foam was reinforced with chopped glass fiber to increase the mechanical properties and fracture toughness of the foam. Fig. 2 shows the fabrication process of the fiber reinforced phenolic foam. The chopped glass fibers were added to the resin mixture containing 20 wt. % of ethanol. The lengths of the chopped glass fibers were 3 and 7 mm, and 0.5, 1.0, 1.5, and 2.0 wt. % were attempted for each length of fiber. The PTSA catalyst content was fixed at 3 wt. % for all of the specimens.

#### 2.4. Tension test of the fiber reinforced phenolic foam

To evaluate the mechanical properties of the fiber reinforced phenolic foam, the tensile strength was measured based on the ASTM standard [19]. The schematic diagram of the tension test specimen is shown in Fig.3(a). The dimensions of the foam were  $25 \times 25 \times 25 \text{ mm}^3$ , and the foam density was  $35 \text{ kg/m}^3$ . The specimen was taken from the middle of the fabricated foam, and the



Fig. 1. Schematic diagram of the process of the microwave foaming method.

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