



# Sustainable nanocomposites of epoxy and silica xerogel synthesized from corn stalk ash: Enhanced thermal and acoustic insulation performance

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## ABSTRACT

The synthesis of silica xerogel from corn stalk ash in ambient pressure drying was carried out by sol-gel method. With the usage of silica xerogel (0.5, 1.0 and 1.5 wt%) in epoxy resin (ER), silica xerogel/epoxy nanocomposites were successfully prepared. The uniform dispersion of silica xerogel in neat ER was observed except nanocomposite including 1.5 wt% silica xerogel. The low density nanocomposites showed high thermal stability and low thermal conductivity. The char residue at 600 °C and thermal conductivity of nanocomposite including 1.5 wt% silica xerogel were specified as 20.30% and 0.220 W/mK, respectively. The acoustic velocity of the nanocomposites was decreased with increasing amount of silica xerogel. The water sorption of the nanocomposites was slightly higher than neat ER. The water contact angle of the nanocomposites were between 75° and 70°. The study provided a new thermal and acoustic insulation material instead of expensive and health risk traditional materials.

## 1. Introduction

The United Nation Environment Program forecasts that the buildings use up approximately 40% of the world global energy, 40% of the global resources and 25% of the global water [1]. And also, the buildings account for more than 30% of the greenhouse gas emissions in many countries [2]. Energy saving thermal insulation materials have attracted a great deal of attention due to the increasing significance of environmental issues in the recent years. Traditional thermal insulation materials such as mineral wool, glass wool, foam glass, expanded polystyrene, extruded polystyrene, phenolic resin foam and polyurethane foam have been used in many applications [3,4]. State-of-the-art thermal insulation materials including vacuum insulation panels, gas-filled panels, aerogels and phase change materials come into prominence with the advantages of space saving, long service life, great thermal performance and high resistance to water and chemicals, lately [5]. At the same time, noise pollution resulted from living activities, railway, urban and highway road traffic, factories etc. is a serious problem all over the world. Nowadays, concrete block, brick, glass, wood, glass wool, foam are generally preferred to decrease sound transmission [6–8].

Silica aerogels are high-performance insulation materials owing to high porosity (~99%), ultralow density (~1 kg/m<sup>3</sup>), low thermal conductivity (0.01 W/mK) and low sound speed (< 70 m/s) [9,10]. The silica aerogels have potential by the help of desirable thermal and

acoustic properties in many applications such as buildings, roofs, refrigerators, refrigerated vehicles, space crafts, vessels, automotive exhaust pipes and so on [11–13]. However, expensive raw materials and complicated drying process with super critical fluids restrict utilization of silica aerogels. Instead of silica aerogels, investigations about synthesis of low cost and environmentally friendly silica xerogels with using inorganic/organic materials or agricultural wastes at ambient pressure drying conditions have rapidly increased [14]. Up to now, various silica based materials are prepared from fly ash [15], oil shale ash [16], kaolin [17], montmorillonite [18], chitosan [19], cellulose [20], bagasse ash [21], sago waste ash [22], wheat starch [23], rice husk [24] for different purposes in the literature.

Epoxy resins (ERs) are one of the commonly used thermoset polymers in structural adhesives, coatings, laminates, construction materials, composites, insulation materials, aerospace structure materials and so on [25–27]. Their properties such as excellent moisture, chemical and solvent resistance, high mechanical strength, low density, low shrinkage, strong adhesive to different substrates, desirable dielectric constant make the ERs promising materials. In spite of these advantages, thermal properties of ERs must be enhanced especially in field required high thermal stability [28,29].

When considering literature studies, there are limited number of studies related with epoxy nanocomposites including silica xerogel obtained from agricultural waste. In this study, low cost silica xerogel was easily synthesized from corn stalk ash at ambient conditions to be

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used as filler in epoxy nanocomposites for the first time. The hopeful results showed that the obtained mesoporous silica xerogel provided to enhance thermal and acoustic insulation performance of the ER.

## 2. Experimental section

### 2.1. Materials

The corn stalk was obtained in dry state from Adana region in Turkey. Sodium hydroxide (NaOH), hydrochloric acid (HCl), isopropanol, n-hexane and distilled water were used in silica xerogel synthesis. Bisphenol A type ER commercially known as NPEL 128 (epoxide equivalent: 184–190 g/eq and viscosity: 12–15 Pa.s at 25 °C) was used as matrix. Methylnadic anhydride and 2,4,6-tris(dimethylaminomethyl) phenol were preferred as hardener and accelerator, respectively.

### 2.2. Pretreatment of corn stalk

The corn stalk was burned to obtain corn stalk ash (CSA) at 650 °C with a heating rate of 5 °C/min in a furnace for 3 h. Acid washing step with 3 M HCl was carried out at 60 °C for 2 h to remove small quantities of minerals. The slurry was filtered and washed with distilled water until pH 7 [30]. Then, the CSA was dried at 70 °C for 24 h in an oven. The chemical composition of corn stalk ash was approximately SiO<sub>2</sub> 49%, CaO 19%, Al<sub>2</sub>O<sub>3</sub> 9%, MgO 8%, Fe<sub>2</sub>O<sub>3</sub> 7%, K<sub>2</sub>O 4%, MnO<sub>2</sub> 2% and Na<sub>2</sub>O 2%.

### 2.3. Synthesis of silica xerogel from CSA

Sol-gel method was efficiently used for synthesis of silica xerogel from CSA. After mixing CSA and 3 M NaOH at a ratio of 1:6 (w/v), the mixture was heated for 5 h at boiling point with constant stirring under reflux. The Na<sub>2</sub>SiO<sub>3</sub> solution was filtered and neutralized by 3 M HCl solution until gelation. Subsequently, the silica gel was washed with distilled water at room temperature three times for 4 h each time. In the aging step to strengthen gel network, the silica gel was immersed in water/isopropanol (v/v: 1/1), isopropanol and n-hexane at 50 °C three times during a day, respectively. Finally, the aged gel was dried at 50 °C for 24 h in an oven [31].

### 2.4. Preparation of silica xerogel/epoxy nanocomposites

The silica xerogels (0.5, 1.0 and 1.5 wt%) and ER were mixed with mechanical stirrer to obtain uniformly dispersed blends for 30 min at room temperature. Removing of entrapped bubbles in each blends and increasing chain mobility to ER were provided by sonication. After adding 30 wt% MNA to blends while stirring, the accelerator was dropped to the each blend. The blends were poured into the mold which was prepared according to the ASTM D 638 Standard. The bubble free blends were cured at 60 °C for 4 h followed by post-curing at 120 °C for 4 h.

### 2.5. Characterizations

Fourier transform infrared spectroscopy (FTIR) analysis was performed on Bruker Vertex 70 to determine chemical bonding state of silica xerogel in the 4000 - 400 cm<sup>-1</sup> range. X-ray diffraction (XRD) pattern was recorded for phase analysis of the silica xerogel by Bruker D8 Advance X-ray diffractometer with Cu K $\alpha$  radiation ( $\lambda = 0.154$  nm) at 40 kV and 40 mA in the range of 10°–80°. N<sub>2</sub> adsorption-desorption isotherm of silica xerogel was obtained on Micromeritics Tristar II 3020 surface area and porosity analyzer at 77 K, after the samples were degassed at 423 K for 12 h. The surface area, average pore diameter and pore volume were determined using Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halende (BJH) method, respectively. Scanning electron

microscopy (SEM) analyses were carried out to investigate morphology of the silica xerogel and epoxy nanocomposites by SM Zeiss LS-10 scanning electron microscope at 20 kV, after coating surfaces with a fine gold layer. Thermogravimetric analyses (TGA) were employed to examine thermal stability of the silica xerogel and epoxy nanocomposites with METTLER STAR SW thermal analyzer at a heating rate of 10°/min. Thermal conductivity of the silica xerogel and epoxy nanocomposites was measured by C-THERM thermal conductivity analyzer at room temperature. The measurements were repeated three times for each material to get reliable results. The pulse-echo method was used to determine acoustic velocity of the epoxy nanocomposites at room temperature [32]. Ultrasonic pulses were supplied at 20 MHz by PAN-AMETRICS-NDT ultrasonic pulse receiver. The measurement was carried out three times on each material and arithmetic average values were specified. The contact angle measurement of epoxy nanocomposites was performed on Dataphysics Oca50Mikro to quantify degree of hydrophobicity. Bulk density of silica xerogel was calculated from mass to volume ratio. The density of each epoxy nanocomposite was calculated using Archimedes method. Water sorption of epoxy nanocomposites was investigated according to ASTM D 570 Standard. The water sorption percentage ( $M_t$ ) was calculated using the following equation:

$$M_t (\%) = \left( \frac{m_t - m_0}{m_0} \right) \times 100 \quad (1)$$

where  $m_t$  and  $m_0$  (g) are mass of the material at time  $t$  and initial, respectively. The water sorption behavior of epoxy nanocomposites was determined using the following equation:

$$\frac{M_t}{M_\infty} = 4 \left( \frac{Dt}{\pi h^2} \right)^{1/2} \quad (2)$$

where  $M_t$  and  $M_\infty$  are water sorption at time  $t$  and equilibrium point, respectively.  $D$  (mm<sup>2</sup>/s) is diffusion coefficient and  $h$  (mm) is thickness of the material.

## 3. Results and discussion

### 3.1. Properties of silica xerogel

The FTIR spectrum of silica xerogel is shown in Fig. 1a. The peak attributed to O-Si-O bending vibration was observed at 459 cm<sup>-1</sup>. While the symmetric stretching vibration of Si-O-Si bond was specified at 798 cm<sup>-1</sup>, the characteristic peak at 1073 cm<sup>-1</sup> was corresponded to asymmetric stretching vibration of Si-O-Si bond [33]. The peak at around 949 cm<sup>-1</sup> indicated Si-OH and Si-O<sup>-</sup> stretching vibrations [34]. The broad band centered at 3600 cm<sup>-1</sup> was related with -OH bond stretching vibration of silanol groups and adsorbed water on the surface. Similarly, it was clearly seen -OH bond bending vibration of adsorbed water at 1644 cm<sup>-1</sup> [20,35]. The bands at 2914 cm<sup>-1</sup> and 1384 cm<sup>-1</sup> were assigned to C-H bonds [36].

The XRD pattern of silica xerogel is shown in Fig. 1b. No significant diffraction peaks were observed of the silica xerogel. The broad peak at around 22° can be explained with amorphous structure formation of silica xerogel [37].

The SEM image of silica xerogel is shown in Fig. 1c. It can be said that the distribution of silica xerogel particles were uniform with nano particle size [38]. The porous structure was ambiguously seen due to low resolution of SEM. And also, it can be expressed with irreversible shrinkage of the silica gel during the aging and ambient pressure drying [16].

The N<sub>2</sub> adsorption-desorption isotherm of silica xerogel is shown in Fig. 1d. The silica xerogel exhibited type IV adsorption isotherm which is characteristic property of mesoporous materials according to IUPAC classification. The hysteresis loop in the desorption cycle was closely associated with capillary condensation in the mesopores [39]. Besides, specific surface area of silica xerogel was determined as high as 252 m<sup>2</sup>/

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