

Advanced fabrication of lightweight aerogels from fly ash for thermal insulation

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ABSTRACT

For the first time, an environmentally friendly and effective procedure to produce high-value engineering aerogels from fly ash (FA) has been developed by dispersing FA particles into a mixture of biodegradable polyvinyl alcohol (PVA) and carboxymethyl cellulose (CMC), followed by freeze-drying. The effect of FA content on the physical properties, morphology, mechanical strength, and thermal conductivity of FA aerogels is also studied comprehensively. The lightweight FA aerogels show a low density of 0.072 – 0.093 g/cm³ with high porosity of 94.94 – 95.78%. The morphology of aerogels shows the uniform distribution of FA particles in PVA-CMC matrixes that creates a porous structure with a pore size of 2-5 μm. Therefore, the FA aerogels exhibit good heat insulation with extremely low thermal conductivity of 0.040 – 0.047 W/m.K at ambient temperature and pressure that is comparable to some commercial insulation materials such as mineral wool, fiberglass, expanded polystyrene, and other silica-based aerogels from waste. Moreover, the compressive modulus of FA aerogels is about 67.73 – 254.75 kPa indicating their excellent mechanical properties under 1 kN vertical compression. The experimental results indicate the significant better durability of FA aerogels than that of previous aerogels from other wastes such as sugarcane bagasse (88 kPa), pineapple leaf fibers (1.64 – 5.34 kPa), recycled polyethylene terephthalate (1.16 – 2.87 kPa), spent coffee grounds (5.41 – 15.62 kPa), and silica – cellulose (86 – 169 kPa). It is concluded that FA aerogels are a promising candidate as a lightweight thermal insulating material.

Key words: Fly Ash, Aerogel, Thermal Insulation, Morphology, Mechanical Strength

INTRODUCTION

Fly ash particles, generated during burning coal in thermal power plants, are one of the reasons causing severe impacts on our ecological environment. Only about 30% of the total FA in Vietnam is recycled into useful products such as concrete, bricks, and insulation panels¹. The remaining FA is piled-up or stored in reservoirs. The surrounding soil, groundwater, and surface water nearby can be contaminated via the percolation of hazardous heavy metals leached from FA. The FA particles can even cause siltation and clog the natural drainage system when they reach to the sub-soil². At the same time, FA particles, which are generally spherical with the size between 10 to 100 μm, are released into the air and become a major source of gas pollution surrounding the dumping sites³.

In recent years, efforts to recycle FA into high-value engineering materials have been carried out to address the pollution of FA and increase the value of this industrial by-product. Among the advanced materials, aerogel is of interest to many scientists due to its extraordinary characteristics such as extremely low density (0.07 – 0.15 g/cm³), high porosity (90 – 99%),

varied surface area (13.8 – 600 m²/g), flexible mechanical properties (0.95 – 150 MPa), and an exceptionally low thermal conductivity which can be down to 0.018 W/m.K⁴. Since the first commercial production, aerogels have been used in many fields, such as aerospace, catalysis, electrical engineering, to name a few⁵.

FA has been used as raw material for synthesizing SiO₂-Al₂O₃ aerogels thanks to its composition including rich SiO₂ (58.9%) and Al₂O₃ (23.9%)⁶. In 2010, Shi et al. fabricated silica aerogels from the industrial FA by ambient pressure drying. The process includes two stages, preparation of silicate solution from FA by hydrothermal reaction with alkali solvent, and fabrication of porous silica aerogels from the obtained solution by sol-gel method. However, before drying, the wet gel was modified by solvent exchange with hydrophobic trimethylchlorosilane (TMCS)/ethanol/hexane solution. The resulting aerogels are lightweight and hydrophobic with a large surface area of 362.2 – 907.9 m²/g⁷. A highly porous silica aerogel could be fabricated by a combination of FA and trona ore which is mainly composed of

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Na₂CO₃ and the ambient pressure drying method¹. To minimize drying shrinkage, the gel obtained after gelation of silicate solution with acid is coagulated with ethanol to form alcogel, after which the alcogel is modified with hydrophobic TMCS/ethanol/hexane solution. The developed aerogel exhibited hydrophobicity and a large specific surface area of 856.2 m²/g. Overall, to synthesize aerogels from FA, the ash is firstly treated with strong alkali to recover silica in the form of silicate solution, followed by a sol-gel route, solvent exchange with hydrophobic solvent, and ambient pressure drying to produce porous aerogels^{1,7}. The synthesis process goes through many steps with prolonged time due to gelation, coagulation, and surface modification happening at room temperature from 12 to 24 hours for each step. With an approach to sustainable development by using less toxic chemicals, shortening synthesis time and applicability in mass production, in this study, FA is dispersed into a viscous solution containing inexpensive and biodegradable polyvinyl alcohol and carboxymethyl cellulose to form a homogenous and stable mixture, followed by gelation at a low temperature of -50 °C before sublimation to remove water under vacuum condition. To the best of our knowledge, no studies have been conducted on the synthesis of FA aerogels by this technique. The FA aerogels generated are comprehensively investigated in terms of their physical properties such as density, porosity, morphology, and potential application in thermal insulation.

MATERIALS AND METHODS

Materials

FA is collected from Duyen Hai Thermal Power Company and separated by a cyclonic system to obtain uniform FA particles. The size of FA before and after separation is 5.71 and 1.37 μm, respectively. The particle distribution and morphology of FA are shown in Figure 1. The chemical composition of FA before and after separation is also tabulated in Table 1. The first binder polyvinyl alcohol (PVA) is purchased from Kuraray Asia Pacific Pte. Ltd (Singapore) with a density ρ_{PVA} of 1.19 g/cm³ and the viscosity of 4.0 wt.% aqueous PVA solution is 22.7 cPs. The second binder carboxymethyl cellulose (CMC) is purchased from Nippon Paper Industries Co., Ltd (Japan) with a density ρ_{CMC} of 1.59 g/cm³. All solution is prepared in distilled water (DW).

Fabrication of FA aerogels

Initially, the mixture of PVA and CMC solution with a solute mass ratio of 2:0.5 is prepared in DW which

is heated to 80 °C. Then, FA with determined weight is dispersed into the PVA/CMC solution by agitating for 3 hours at 80 °C to form a homogenous mixture. After that, the mixture is agitated for another 30 minutes at room temperature before being frozen at -50 °C within 4 hours for gelation. Finally, the sample is freeze-dried under vacuum condition for 48 hours to obtain lightweight FA aerogel. The FA aerogels are fabricated with different FA contents of 3.0, 4.0, and 5.0 wt.%.

Characterization

The density of FA aerogels is determined by measuring their weight and volume via diameter and thickness. The porosity (φ) of the aerogels is calculated by (Eq. 1) based on their density (ρ_a) and average densities of components (ρ_b).

$$\phi = \left(1 - \frac{\rho_a}{\rho_b} \right) \times 100 \quad (1)$$

$$\rho_b = \frac{C_{FA} + C_{CMC} + C_{PVA}}{\frac{C_{FA}}{\rho_{FA}} + \frac{C_{CMC}}{\rho_{CMC}} + \frac{C_{PVA}}{\rho_{PVA}}} \quad (2)$$

where C_{FA}, C_{PVA}, and C_{CMC} are the content of FA, PVA, and CMC, respectively. ρ_{FA} that is the density of FA powder after separation is about 2.26 g/cm³.

Morphology of FA aerogels is characterized by field emission-scanning electron microscopy (FE-SEM) Hitachi S4800. The specimens are coated with a thin layer of Pt to enhance the resolution of the images captured.

The thermal conductivity of the prepared aerogels is measured by TCi C-Therm Thermal Conductivity Analyzer (Fredericton, NB, Canada) with the modified transient plane source method. The average value is obtained after three measurements for each sample at ambient temperature.

The thermal stability of FA aerogels is investigated by thermogravimetric analysis by LabSys Evo TG/DSC 1600 Setaram in air. The samples are heated from room temperature to 800 °C with the heating rate of 10 °C/min. The change in weight of specimens following the temperature is recorded.

The compressive strength of the fabricated aerogels is determined by Instron 5500 (Norwood, USA). During the test, the specimens are under a loading rate of 1.0 mm/min.

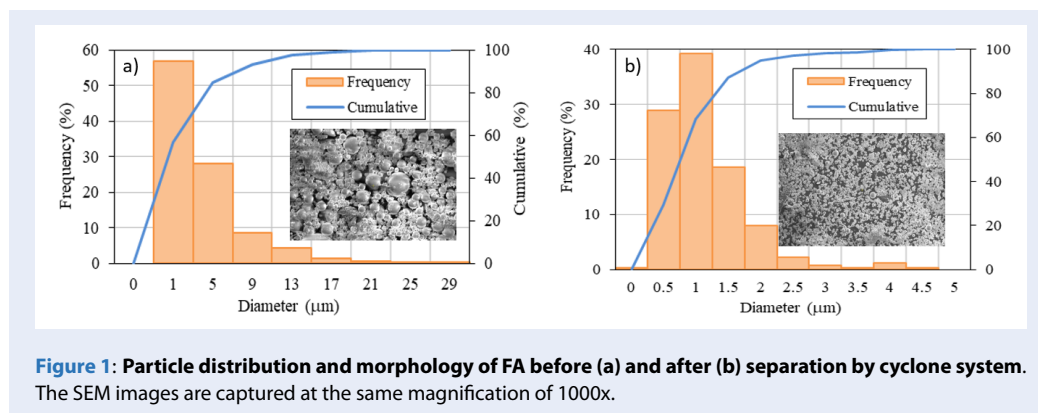


Figure 1: Particle distribution and morphology of FA before (a) and after (b) separation by cyclone system. The SEM images are captured at the same magnification of 1000x.

Table 1: Chemical composition of FA before and after separation

Sample	Content (wt.%)				LOI* (%)
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Others	
RFA	55.07	19.10	7.25	18.58	13.0
SFA	59.58	27.31	5.93	7.18	2.0

*LOI: Loss of ignition

RESULTS AND DISCUSSION

Morphology of FA aerogels

A combination of FA particles which are separated by cyclone and green binders including PVA and CMC successfully generates FA aerogels (Figure 2a) by eco-friendly and cost-effective freeze-drying method. The sublimation of ice, which is formed at the gelation step, leaves small holes inside the aerogels as can be seen from Figure 2b-d. These holes are interconnected and irregular because the freezing process occurs naturally leading to diversity in ice crystal sizes. However, as the network of pores is created, the FA aerogels contain a lot of air making them lightweight with an extremely low density of 0.072 – 0.093 g/cm³ as shown in Table 2. Moreover, with increasing FA content, the aerogels become denser because of fewer air pockets and a decrease in pore size. It is predicted that the porosity of FA aerogels also decreases.

Because of the porous network inside, the fabricated aerogels have a high porosity of 94.94 – 95.78%. As higher FA is used, a decrease in porosity of the aerogel is witnessed because of an increase in the number of particles taking up space inside the aerogel. In contrast, the density of the FA aerogel increases since more and more FA particles are presented in the same unit volume of material. Compared to previous silica aerogels from FA via sol-gel route and ambient drying by Qin et al. (0.0915 – 0.2379 g/cm³) and Wu et al. (0.099 g/cm³), the FA aerogels in this work exhibit

much lower density^{1,8}. Furthermore, the procedure of synthesizing our FA aerogels uses no alkaline solvents for pretreatment of FA to obtain pure silicate and expensive organic chemicals for surface modification of silica gel before ambient drying as published in previous studies. Hence, the fabrication of FA aerogels saves raw materials cost, is more environmentally friendly and the total synthesis time is reduced 20% compared to the previous works^{1,8}.

Thermal insulation of FA aerogels

The potential heat insulation of FA aerogels is evaluated by their thermal conductivity which is measured at ambient temperature (25 °C). Overall, the FA aerogels show low thermal conductivity of 0.040 – 0.047 W/m.K at 24.1 °C, indicating their excellent heat insulation (Table 3). The heat transfer through aerogel is contributed by the conduction of the solid-state network, conduction of gas phase, and radiation within pores⁹. Air is one of the excellent thermal insulators due to its extremely low thermal conductivity of 0.026 W/m.K¹⁰. Combining the outstanding thermal insulation properties of air and the high porosity of aerogels above 94%, the air captured inside is the main reason why the aerogels have such low thermal conductivity below 0.05 W/m.K.

With increasing FA content from 3.0 to 5.0 wt.%, the porosity of FA aerogels decreases causing the efficiency of gas-phase conduction to decrease, and thus, their thermal conductivity increases from 0.040 to

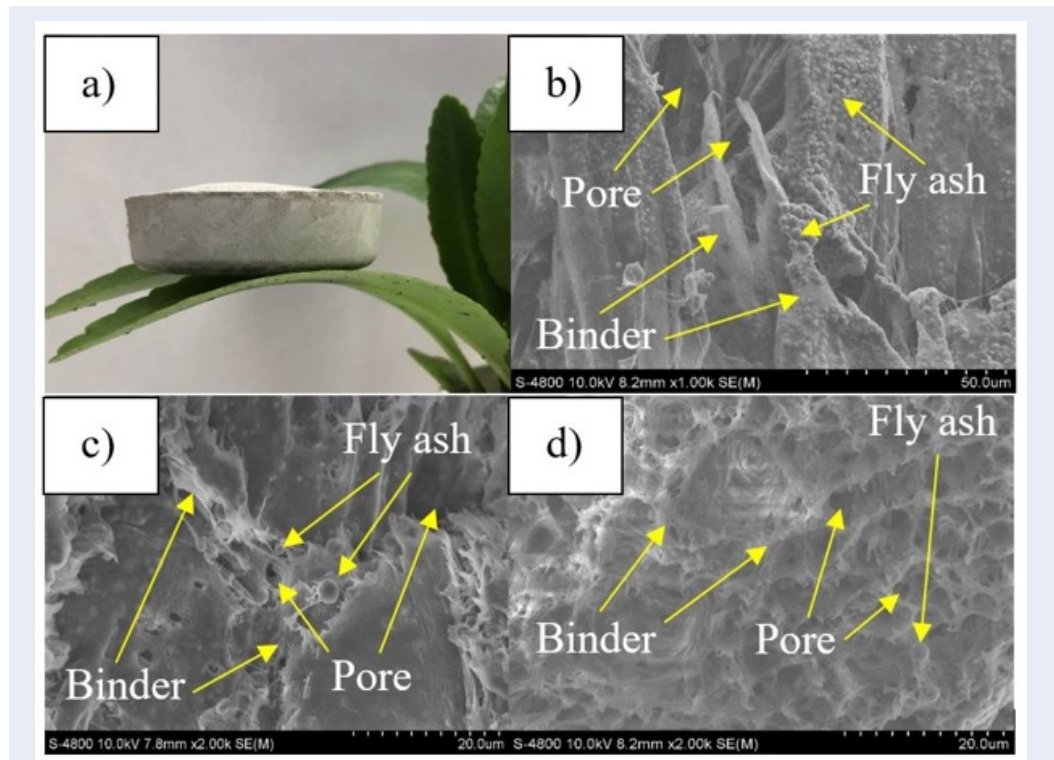


Figure 2: A photograph of ultra-lightweight FA aerogel (a) and SEM images of FA aerogels with different FA contents: (b) 3.0 wt.%, (c) 4.0 wt.%, and (d) 5.0 wt.%.

Table 2: Summary of density and porosity of fabricated FA aerogels

Sample	FA content (wt.%)	Density (g/cm ³)	Porosity (%)
FAA1	3.0	0.072 ± 0.003	95.78 ± 0.15
FAA2	4.0	0.083 ± 0.002	95.35 ± 0.12
FAA3	5.0	0.093 ± 0.004	94.94 ± 0.24

Table 3: Thermal conductivity and compressive modulus of FA aerogels

Sample	Thermal conductivity (W/m.K)	Young's modulus (kPa)
FAA1	0.040 ± 0.001	67.73 ± 0.14
FAA2	0.044 ± 0.001	157.63 ± 0.08
FAA3	0.047 ± 0.002	254.75 ± 1.00

0.047 W/m.K. Moreover, the thermal conductivity of our FA aerogels is comparable to some commercial insulation materials such as mineral wool (0.030 – 0.040 W/m.K), fiberglass (0.033 – 0.044 W/m.K), expanded polystyrene (0.030 – 0.040 W/m.K) and other silica aerogels from gold mine (above 0.2 W/m.K), coal gangue (0.026 W/m.K), and dislodged sludge (0.030 – 0.087 W/m.K)¹¹⁻¹⁴. It can be claimed that the aerogels from FA are a promising candidate for practical heat insulation applications.

Thermal stability of FA aerogels

Regarding the TGA results presented in Figure 3a, a gradual increase in FA concentration plays an impact on the thermal stability of FA aerogels. In the atmospheric condition containing air, the FA aerogels exhibit two main phases of mass change by the temperature as follows: (i) 80 – 100 °C and (ii) 250 – 475 °C. The weight loss of 3 – 5% in all samples at the first stage is due to the evaporation of water adsorbed into

the aerogels because of hydrophilic hydroxyl groups on PVA and CMC matrixes. From 100 to below 250 °C, the weight of FA aerogels remains unchanged indicating that the materials can withstand the temperature of up to 250 °C.

However, a significant weight loss of about 40% is witnessed in the next period as the temperature gradually increases to 475 °C. This thermal decomposition of FA aerogels is because of the degradation of binders including PVA and CMC with the decomposition temperature of 250 and 350 °C, respectively, and the decomposition of CaCO₃ and residual coal of original coal in FA¹⁵. According to the DTA graph in Figure 3b, the thermal decomposition of FA aerogels is an exothermic process because the DTA curves have upward peaks, in which the FAA1 gives the highest heat released of nearly 40 μV at 430 °C.

At higher temperatures from 500 to 800 °C, the weight of all samples is kept constant since the specimens then consist of only FA and ash resulting from the previous oxidative degradation of PVA and CMC. Although all samples have the same initial decomposition temperature, FAA3 exhibits the highest remaining weight percentage of about 62% at 500 °C because its original FA content is the highest among the tested concentrations.

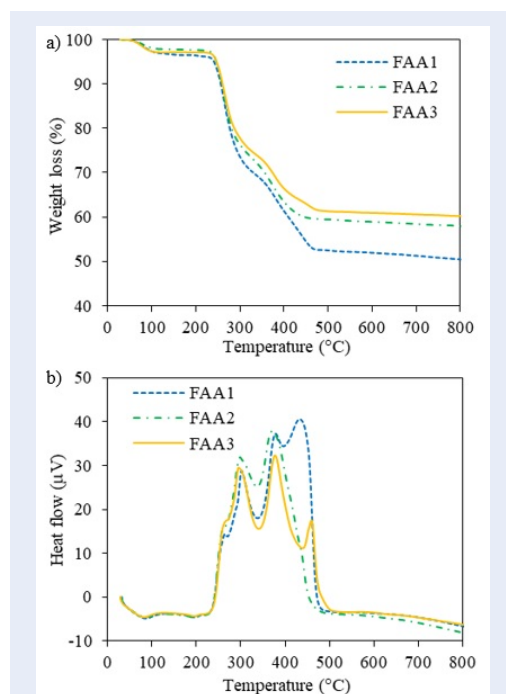


Figure 3: TGA (a) and DTA (b) patterns of FA aerogels at various FA concentrations of 3.0, 4.0, and 5.0 wt.%.

Mechanical strength of FA aerogels

The results of the compressive strength of FA aerogels are summarized in Table 2 and Figure 4. As increasing FA content from 3.0 to 5.0 wt.%, Young’s modulus of aerogels increases from 67.73 to 254.75 kPa, indicating their better durability under loading of 1.000 N than previous aerogels from wastes such as sugarcane bagasse (88 kPa), pineapple leaf fibers (1.64 – 5.34 kPa), recycled polyethylene terephthalate (1.16 – 2.87 kPa), spent coffee grounds (5.41 – 15.62 kPa), silica – cellulose (86 – 169 kPa)¹⁶⁻¹⁹. At the first 10% of strain known as linear elastic region, the stress increases with increasing FA content, in which that for aerogel composed of 5.0 wt.% is highest²⁰. However, the sample containing 4 wt.% FA has the same stress at 10% strain as the one having 5 wt.% FA. This may be because the porous structure of FAA2 collapses then, causing a considerable increase in the compressive stress.

CONCLUSION

For the first time, FA has been successfully converted into durable and thermal insulation aerogels by using eco-friendly binders such as polyvinyl alcohol and carboxymethyl cellulose and freeze-drying technique. The fabricated FA aerogels are lightweight with low density and high porosity since the porous structure is formed after sublimation of distilled water without any damage. A special feature of FA aerogels in this work is their outstanding compressibility over previous aerogels which have been synthesized before. Therefore, FA aerogel is demonstrated to be a promising candidate for heat insulation in practice.

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ABBREVIATION

- BET: Brunauer–Emmett–Teller
- CMC: Carboxymethyl cellulose
- DTA: Differential thermal analysis
- DW: Distilled water
- FA: FA
- LOI: Loss of ignition
- PVA: Polyvinyl alcohol
- RFA: Raw FA

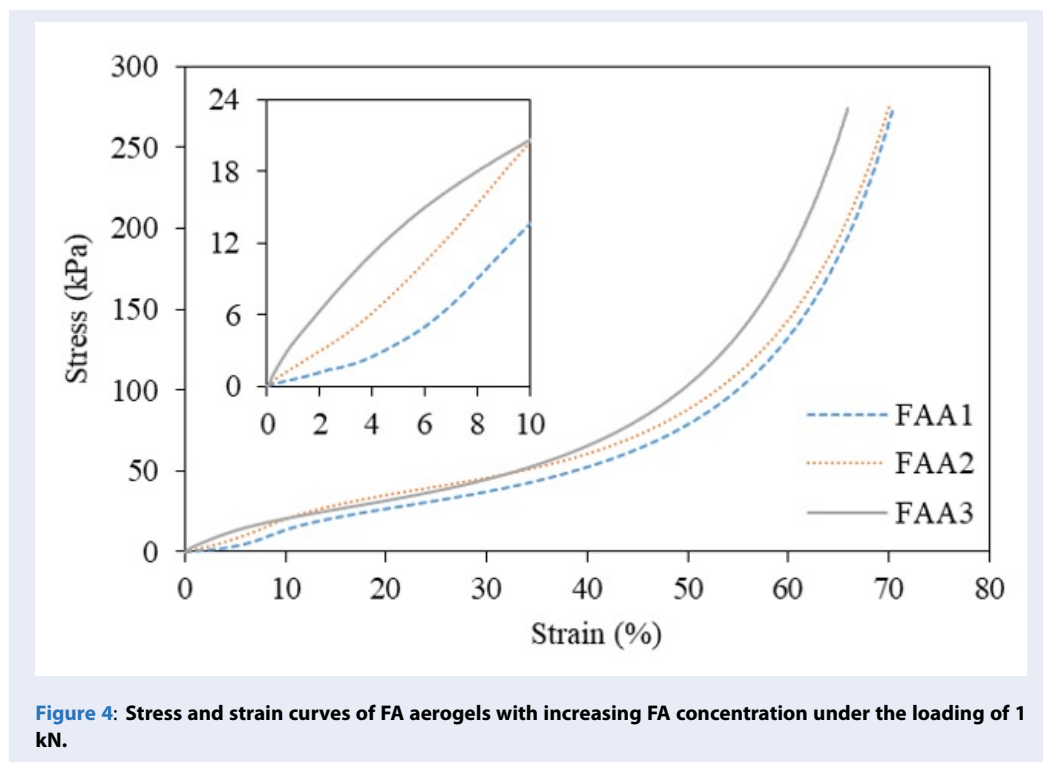


Figure 4: Stress and strain curves of FA aerogels with increasing FA concentration under the loading of 1 kN.

SEM: Scanning electron microscopy
 SFA: Separated FA
 TGA: Thermogravimetric analysis
 TMCS: Trimethylchlorosilane

COMPETING INTERESTS

The authors declare that they have no competing interests.

AUTHORS' CONTRIBUTIONS

Nga Hoang Nguyen Do: Conceptualization, Visualization, Writing – original draft. **Huy Gia Tran:** Methodology, Investigation, Formal analysis. **Huong Ly Xuan Doan:** Characterization, Data analysis. **Nghiep Quoc Pham:** Data curation, Resources. **Kien Anh Le:** Validation, Writing – review & editing. **Phung Kim Le:** Writing – review & editing, Funding acquisition, Project administration.

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Tổng hợp aerogel siêu nhẹ từ tro bay định hướng cách nhiệt

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TÓM TẮT

Lần đầu tiên một quy trình thân thiện môi trường và hiệu quả để tổng hợp aerogel có giá trị kỹ thuật cao từ tro bay (FA) được xây dựng hoàn chỉnh bằng cách phân tán các hạt FA vào hỗn hợp chất kết dính polyvinyl alcohol (PVA) và carboxymethyl cellulose (CMC) có khả năng phân hủy sinh học và phương pháp sấy thăng hoa. Ảnh hưởng của hàm lượng FA đến các tính chất vật lý, hình thái học, độ bền cơ học và độ dẫn nhiệt của FA aerogel cũng được nghiên cứu. Vật liệu FA aerogel siêu nhẹ được tổng hợp có khối lượng riêng thấp (0,072 – 0,093 g/cm³) với độ xốp lớn (94,94 – 95,78%). Hình thái học của FA aerogel thể hiện sự phân bố đồng đều của các hạt tro bay trong mạng lưới liên kết giữa PVA và CMC hình thành nên cấu trúc rỗng xốp với kích thước lỗ rỗng là 2-5 mm. Vì vậy, FA aerogel có tính chất cách nhiệt tốt với độ dẫn nhiệt rất thấp (0,040 – 0,047 W/m.K) ở nhiệt độ phòng và áp suất thường. Bên cạnh đó, độ dẫn nhiệt của tro bay aerogel tương đương với các vật liệu cách nhiệt thương mại như bông khoáng, bông thủy tinh, polystyrene và các loại silica aerogel khác từ chất thải. Thêm vào đó, mô-đun nén của tro bay aerogel từ 67,73 – 254,75 kPa, chứng tỏ đặc tính cơ học nổi trội của vật liệu dưới lực nén 1 kN theo phương thẳng đứng. Kết quả thí nghiệm cho thấy độ bền cơ học của tro bay aerogel cao hơn so với các aerogel từ chất thải khác như bã mía (88 kPa), sợi lá dứa (1,64 – 5,34 kPa), sợi polyethylene terephthalate tái chế (1,16 – 2,87 kPa), bã cà phê (5,41 – 15,62 kPa), silica – cellulose (86 – 169 kPa). Như vậy, FA aerogel là vật liệu cách nhiệt siêu nhẹ đầy tiềm năng.

Từ khóa: Tro bay, Aerogel, Cách nhiệt, Hình thái học, Độ bền cơ học

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