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In situ preparation of intrinsic flame retardant urea formaldehyde/aramid fiber composite foam: Structure, property and reinforcing mechanism



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ARTICLE INFO	A B S T R A C T			
Keywords: Urea-formaldehyde (UF) foam Aramid fiber Intrinsic flame retardancy Reinforcing mechanism	The intrinsic flame-retardant urea-formaldehyde/aramid fiber (UF/AF) composite foam was fabricated via in situ water-based foaming process. At low AF content (< 3 wt%), AF can disperse homogeneously in foam and act as heterogeneous nucleating agent, resulting in smaller cell size, narrower cell size distribution, lower porosity. For UF/2 wt% AF foam, the pulverization ratio decreased by 11%, while the compressive strength and modulus increased by 198% and 257% respectively. As the "pillar" structure, AF enhanced the stiffness of the cell wall, while under compressive stress, AF can play a role of bridge between the cracks, preventing further expansion of the crack, increasing the crack propagation path and thus delaying the disastrous collapse of cells. By addition of AF, the decomposition temperature and char yield increased, while the UL-94 tests were classed as a V-0 rating, and during burning, the expansion of the flame was suppressed remarkably for the composite foams.			

1. Introduction

As a typical kind of thermosetting amino resin foam, urea-formaldehyde (UF) foam is manufactured through the environmental friendly water-based foaming process by the multifold reaction of the two monomers of urea and formaldehyde [1–4]. Due to the carbon/ nitrogen enriched structure, when UF foam burns, inert gases such as NH₃, CO₂ etc. are generated which can dilute oxygen, and meanwhile, a carbon layer forms to isolate the combustible gases and prevent the expansion of the flame. Thus, UF foam exhibits outstanding intrinsic flame retardancy and non-dripping during combustion [5,6], which is superior to that of widely used thermoplastic foams, such as polyurethane, polystyrene and polyethylene foam with low heat distortion point and high combustibility [7–12]. Moreover, UF foam possesses the advantages of low cost, and shows a promising future in the fields of building insulation, electronic appliances, chemical industry and aerospace application [13].

However, due to the lack of flexible functional groups in UF molecules, UF foam suffers from high brittleness, high pulverization ratio and low strength, and thus, its applications are greatly restricted [14,15]. At present, few literatures are available on improving the strength and toughness of UF foam. Shen prepared polyvinyl alcohol toughened UF foam, and the compressive strength of the foam increased from 50 KPa for the neat foam to 215 KPa for the modified sample [16]. In our previous study, melamine (MA) was chemically introduced into UF system via co-condensation reactions, and the ureamelamine-formaldehyde (UMF) foam with elastic compressive behaviour was obtained [17]. Moreover, UF/grapheme nano-composite foam had also been prepared in our group, which showed that, by introduction of 0.5 wt% GN, the compressive strength and modulus increased by 71.8% and 132.6%, respectively [18].

Aramid fiber (AF) is a kind of man-made high-performance fiber with molecules chains mainly composed of aromatic rings and amide groups. Owing to the high crystallization and orientation of its macromolecular structure, AF shows superior properties, such as high strength-to-weight ratio (about five times higher than steel) and excellent heat resistance [19-21]. Therefore, AF has been utilized as an ideal reinforcement for polymer-based foams. In this study, the intrinsic flame-retardant UF/AF composite foam was fabricated via in situ waterbased foaming process for the purpose of reinforcing UF foam. Compared with other high performance fibers (e.g. poly-p-phenylene benzobisoxazole fibers [22-24] and quartz fibers [25]), a large amount of amide groups on the AF chains make it possible to generate chemical bonding and hydrogen bonding interactions with hydroxyl and primary/secondary amino groups of UF matrix, which was beneficial to enhance the compatibility of the two phases. Moreover, through in situ preparation, better dispersion of AF in UF pre-polymer resin can be achieved, and then during the curing process, this ideal dispersion state

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Received 23 July 2018; Received in revised form 1 October 2018; Accepted 8 October 2018 Available online 09 October 2018 1359-835X/ © 2018 Published by Elsevier Ltd. can be fixed in the composite foam. The effect of AF content on the cellular structure, compressive mechanical behavior, thermal stability and flame retardancy of UF foam was studied, in order to obtain UF foam with intrinsic flame-retardancy and high strength. Moreover, the reinforcing mechanism of UF/AF composite foams was explored.

2. Experimental

2.1. Materials

Urea was provided by Bodi Chemical Co. Ltd (Tianjin, China). Formaldehyde water solution (37 wt%) as analytically pure reagents was purchased from Jinsan Chemical Reagent Co. Ltd (Chengdu, China). Sodium hydroxide (NaOH) was obtained from Xilong Chemical Co. Ltd (Chengdu, China). Formic acid, sodium dodecyl benzene sulfonate (SDBS), triethanolamine and pentane were all provided by Aike Chemical Regents Factory (Chengdu, China). Short aramid fibres with length of 3 mm were provided by China Bluestar Chengrand CO. Ltd. (Chengdu, China).

2.2. Synthesis and preparation

2.2.1. Synthesis of UF pre-polymer resin

UF pre-polymer resin was synthesized with the traditional alkalineacid-alkaline three-step reactions [26,27]. A proper quantity of urea and formaldehyde (mole ratio of the formaldehyde to urea was 1.7:1.) was put into the reactor. The mixture was adjusted to pH 7.8 with 10 mol/L NaOH aqueous solution and then heated to 75 °C, maintaining for 50 min with vigorous stirring. Subsequently, the system was adjusted to acidic conditions and the second part of urea was added. Finally, the system was adjusted to pH 7.8 again and the resultant UF resin was obtained.

2.2.2. Preparation of UF/AF foam

At first, AF and UF pre-polymer resins were homogeneously blended and the mixture was compounded with emulsifier (SDBS) and foaming agent (pentane) by a high-speed mechanical mixer. Then curing catalyst (formic acid) was added and stirred quickly for 40 s. Finally, the obtained viscous mixture was quickly poured into a foaming mould, and then cured and foamed in the oven. The preparation process of UF/AF composite foam was depicted in Fig. 1 and the detailed formulations were listed in Table 1.

2.3. Measurements

2.3.1. Scanning electronic microscopy (SEM) analysis

The micro-porous morphology of UF/AF foam was observed with a JSM-5900LV SEM (JEOL Ltd. Japan) under operating voltage of 5 kV. The samples were ion beam sputter-coated with gold and the thin layer thickness was about 1-20 nm. The cell size and its distribution were statistical analysed with Image Pro Plus 6.0 software [28,29].

Table 1				
The formulations	for	UF/AF	composite	foam.

Sample	AF (wt%)	SDBS (wt%)	Pentane (wt%)	Formic acid (wt%)
UF	0	1	3	5
UF/0.5 wt%AF	0.5	1	3	5
UF/1.0 wt%AF	1.0	1	3	5
UF/2.0 wt%AF	2.0	1	3	5
UF/3.0 wt%AF	3.0	1	3	5
UF/3.0 wt%AF	3.0	1	3	5

2.3.2. Apparent density

The apparent density of UF/AF foam was measured according to ISO 845:2006, and calculated as follows [30]:

 $\rho = m/v$

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where, ρ is the apparent density (kg m⁻³) of the sample; *m* is the mass (kg) and *v* is the volume of the sample (m³). Samples with 50 × 50 mm in squares and 10 mm in thickness were prepared.

2.3.3. Water absorption and porosity

The water absorption of the UF/AF foam was investigated according to ISO 2896:2001. Samples with dimensions of $50 \text{ mm} \times 50 \text{ mm} \times 20 \text{ mm}$ were dried in the oven for 2 h at first, and then were immersed into the distilled water for 24 h. Finally, samples were taken out and wiped to remove the excessive water on their surface. By measuring of the weight changes of the samples before and after immersion, the water absorption can be calculated as follows [29]:

Water absorption(%) = $[(m_2 - m_1)/m_1] \times 100\%$

where m_1 is the mass of dry sample before immersion and m_2 is the mass of foam after immersion in the water for 24 h.

The porosity of the UF/AF foam was also measured with an immersion method according to GB10799-2008, and determined with the following formula [29,31]:

$$Porosity = [(G_2 - G_1)/\rho] / [(G_2 - G_3 + G_4)/\rho] = [(G_2 - G_1)] / [(G_2 - G_3 + G_4)]$$

where G_1 is the weight of the dry foam in the air (before immersion); G_2 is the weight of saturated foam after immersion in the water for 24 h; G_3 is the total weight of the saturated foam and tray measured in the water; G_4 is the weight of the tray measured in the water; ρ is the density of water.

2.3.4. Compression properties

According to ISO 844:244, the compressive test was carried out with a 4302 material testing machine (Instron Co., USA.). The foams were compressed to 50% of the initial thickness. Parallel tests were made at least five times [29].

2.3.5. Thermogravimetric analysis (TGA)

Thermogravimetric analysis was performed under nitrogen flow on a STD Q600 thermo-analyzer instrument (TA Co., USA) at a heating rate of 10 °C min⁻¹. Samples were heated from 20 °C to 700 °C.



Fig. 1. Schematic diagram of the fabrication process of UF/AF foam.

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