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ARTICLE

Allyl Glycidyl Ether-modified Animal Glue Binder for Improved Water Resistance and Bonding Strength in Sand Casting

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ABSTRACT

This paper aims to develop a modified animal glue sand binder for foundry casting with improved water resistance and bonding strength. An efficient method is reported by using sodium hydroxide as the catalyst to improve the operability of animal glue binder and allyl glycidyl ether as the modifier to improve the water resistance and bonding strength. Sand specimens prepared using allyl glycidyl ether-modified animal glue binder were cured by compressed air at room temperature. The proposed method saves energy and is environmentally friendly and inexpensive. Compared with unmodified animal glue binder, standard dog bone sand specimens with allyl glycidyl ether-modified animal glue binder had higher tensile strength of 2.58 MPa, flowability of 1.95 g, better water resistance (a lower decrease in tensile strength at 25 °C and relative humidity of 60%), and good collapsibility. This allyl glycidyl ether-modified animal glue binder is suitable for practical application in the foundry industry.

1. Introduction

Foundries play an important industrial role [1]. A key technical component is the casting binder. Organic and inorganic binders are extensively used in the foundry industry [2-9]. However, pyrolysis of organic substances from these binders emits pollutant gases that are harmful to people and the environment. In comparison with organic binders such as phenolic urethane, inorganic binder such as sodium silicate has low content of organic substances and is recognized as the most environmentally friendly foundry binders [10-12]. But the silica gel has lower strengths after curing, therefore a large ratio of water glass/sand is required, this would cause lower flowability [7]. CO_2 and ester hardened sodium silicate binder has poor collapsibility, and microwave hardened sodium silicate binder has a large hygroscopicity and high requirement of casting mold. To meet the increasing environmental demands, seeking environmentally friendly high-performance binders is still an urgent issue. In recent years, researchers reported the use of natural raw material-based binders derived from plants, such as starch and its derivatives [13-19] and plant protein [20]. Modified starch can be used as a binder in the foundry industry [13,21-23]. $NaHSO_3$-modified soy protein had better adhesion performance and water resistance than traditional soy protein isolate adhesive [20].

Animal glue is derived from renewable natural sourc-

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es such as animal's skin, bone, and tendon. Animal glue mainly contains various types of polypeptide molecules, or long chains of amino acids [24]. It is non-toxic, biodegradable, and environmentally friendly; however, it cannot be directly used as a binder in the foundry industry due to its poor mechanical properties, poor water resistance, and low operability. Liu et al [25]. prepared an animal glue binder using acrylic acid, ammonium persulfate, and glucose at a mass ratio of 30:3:15 as a modifier, and obtained a final tensile strength of 3.36 MPa with the addition of 3% binder to sand; however, heat curing at high temperature (180 ºC for 20 min) was required, which is highly energy-consuming and may cause oxidative decomposition of the proteins. Liu et al. [26] used glycerin, glycol, and dextrin at a mass ratio of 9:16:15 to modify animal glue, and cured experimental sand samples by blowing high-pressure carbon dioxide gas at a flux of 0.7 $m^3 \cdot h^{-1}$ for 60 s. The original and final (24 h) strengths of the standard dog bone sand specimens were 0.7 and 4.2 MPa, respectively. For this binder, the original strength was low, a long time (24 h) was required to reach a suitable tensile strength, and a study of the water resistance of the standard dog bone sand specimens was needed.

The present study aimed to develop a novel modified animal glue binder having advantages of good mechanical properties, good water resistance, low cost, environmentally friendly characteristics, and low energy consumption by using allyl glycidyl ether as the modifier. Allyl glycidyl ether was expected to react with active hydrogens of hydrolyzed animal glue, thus improved its water resistance. Experimental standard dog bone sand specimens were cured by blowing air.

2. Materials and Method

Animal glue was obtained from Hubei Jusheng Technology Co. (China). Allyl glycidyl ether (analytical reagent (AR), 99%), NaOH (AR, ≥ 96%), and $Ca(OH)_2$ (AR, ≥ 95%) were purchased from Aladdin Co. (China). Standard sand (ZGS-50/100(60) grade) was received from Chongqing Changjiang Modeling Materials Group Co. (China).

The allyl glycidyl ether-modified animal glue binder was prepared according to previous methods with some modifications [24,25]. Animal glue (50 g) and distilled water (45 g) were placed in a 250-mL three-necked flask equipped with a mechanical stirrer. The mixture was kept at room temperature for 10 min until the solution was sufficiently swollen, heated to 40 °C and reacted for 30 min, and then heated to 50 °C and 40 mass% NaOH (5 mL) added dropwise as the catalyst. After hydrolysis for 30 min, allyl glycidyl ether (0.75 g) was added into the mixture as the modifier and allowed to react for 90 min. The reaction system was cooled to room temperature and approximately 100 g allyl glycidyl ether-modified animal glue binder was obtained as a brown liquid. A similar procedure was used to prepare unmodified animal glue binder that contained no modifier as a control.

Viscosity of the allyl glycidyl ether-modified animal glue binder was measured using an NDJ-4 rotary viscometer (Shanghai Precision Science Instrument Co., China) at H grade, 60 $rpm \cdot min^{-1}$, and 25 °C.

Approximately 1.0 g modified or unmodified animal glue binder was placed in a crucible and dried at 105 °C to constant mass. The resultant solid was ground to powder with a mortar and subjected to elemental, thermogravimetric (TGA), and Fourier transform infrared (FTIR) analyses using an Elementar Vario Micro Select instrument (Germany), TGA209F1 thermogravimetric analyzer (NETZSCH, Germany), and Spectrum 400 spectrometer (Perkin-Elmer, USA) over the 4000−400 cm^{-1} region, respectively.

Anhydrous calcium hydroxide (8 g) and standard sand (1000 g) were mixed with stirring for 15s. Allyl glycidyl ether-modified animal glue binder (40 g) was then added and coated sand was obtained by further mixing with stirring for 90s. The uniformly coated sand was placed in the core shooting funnel for a core shooting time of 5s and core shooting pressure of 0.5 MPa. Then the coated sand was placed into the 8-shape mold. The standard dog bone sand specimens was prepared by curing the coated sand using a core shooting machine (OuNai Mechanical Mold Co., China) at a compressed air pressure of 0.5 MPa and blowing time of 5 min. The samples were removed for characterization. All tests were repeated three times and averaged values are reported. Tensile strength of the standard dog bone sand specimens was determined according to AFS Test Procedure 3301-00-S, as per the AFS Mold and Core Test Handbook [27] using an SWY tensile strength testing machine (Chongqing Changjiang Modeling Materials Group Co., China). Figure 1 shows the dog bone specimen and core box.

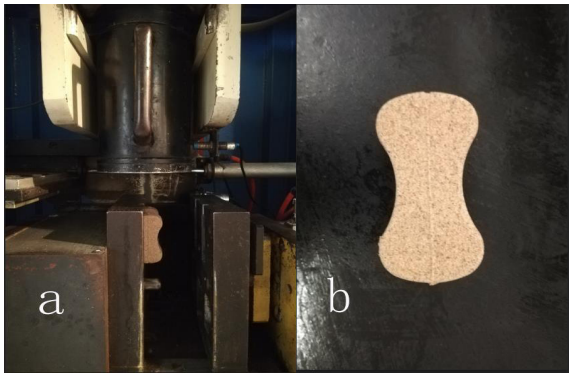

Figure 1. The core box(a) and dog-bone specimen (b)

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Flowability of the coated sand was tested according to the Machinery Industry Standards of China (Title: Wet inorganic binder coated sand for nonferrous metal foundry; Standard No: JB/T 13082-2017). The test procedure was described as follows. A hole of diameter 12 mm (height from the center of the cylinder to the bottom of the sample tube was 16 mm) was opened on the side of a 50 mm diameter sample tube (smooth inner wall). Prior to a test, the hole was plugged with a plunger and 185 g of coated sand placed into the sample tube. The plunger was then pulled out and the coated sand hammered 10 times. The combined mass of sand left in the hole and the extruded grit was used to characterize the flowability of the coated sand. Figure 2 shows equipment for measuring the flowability of the coated sand.

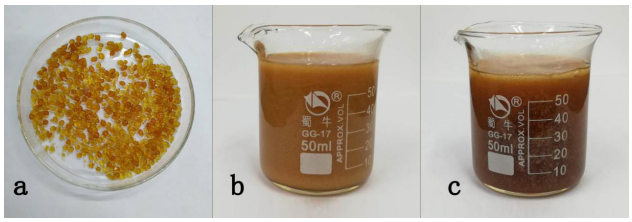
Figure 2. The equipment for measuring the flowability of the coated sand

Water resistance was evaluated by placing the samples in a constant-temperature and humidity chamber at 25 °C and relative humidity of 60%, then removing them for tensile strength measurements after different times. To determine core collapsibility, the standard dog bone sand specimens were kept at 400, 500, and 600 °C for 5 min and then cooled to room temperature and their tensile strength measured. Gas production was measured at 850 ºC using an SFL gas evolution test apparatus (Beijing Jiatian Foundry Material Technology Co., China) using an accurately weighed (1 ± 0.01 g) finely divided sand specimens.

3. Results and Discussion

Animal glue is a high-molecular-mass polymer with a particularly complicated spatial structure in solid state (Figure 3a), so it is not suitable for use as a binder in the foundry industry due to its large intrinsic viscosity and poor fluidity. Animal glue is hydrolyzed to reduce its viscosity using bases, such as NaOH, KOH, and $Ca(OH)_2$, as the catalyst. Of these, NaOH was reported to perform best [25]; therefore, NaOH was selected as the catalyst and the effect of NaOH amount on the tensile strength of standard dog bone sand specimens was investigated (Figure 3d). The tensile strength of standard dog bone sand

Specimens increased from 0.38 to 1.98 MPa as the NaOH content increased from 0.0 to 4.0 mass%, based on the solid content of animal glue binder. The tensile strength decreased to 1.32 and 0.72 MPa as the NaOH content was further increased to 5.0 mass% and 6.0 mass%, respectively. This indicated that 4.0 mass% of NaOH was the optimum catalyst value. As NaOH was added to raw animal glue, the amide bonds in the glue were progressively hydrolyzed and its spatial structure was destroyed, resulting in a low molecular mass and viscosity. At 4.0 mass% NaOH, the molecular mass and viscosity of the modified animal glue were optimum for its use as a sand binder. Further increase of NaOH caused excess hydrolysis of the glue, resulting in further lowering of the molecular mass and viscosity, which led to decreased tensile strength of the standard dog bone sand specimens.



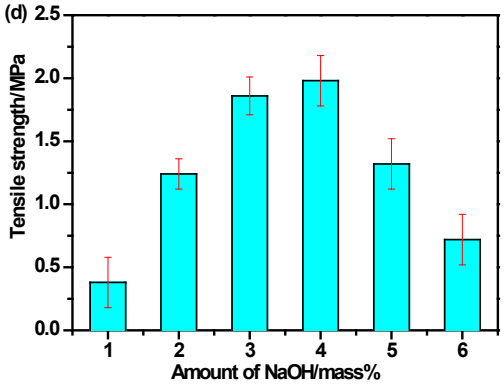


Figure 3. The morphology of raw animal glue (a), unmodified animal glue binder (b), and modified animal glue binder (c). Effect of mass ratio of NaOH on tensile strength of standard dog bone sand specimens (d)

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The hydrolyzed animal glue is a brown liquid (Figure 3b) which possesses many active hydrogens, which would cause poor water resistance if directly used as a binder in the foundry industry. Allyl glycidyl ether, selected here as the modifier, was expected to improve water resistance by reacting with the active hydrogens of the hydrolyzed animal glue. Allyl glycidyl ether modified animal glue exhibits a dark brown color (Figure 3c). The effect of allyl glycidyl ether content on the strength of standard dog bone sand specimens was studied. For a modification time of 1.0h, the tensile strength of standard dog bone sand specimens increased from 1.93 to 2.20 MPa as the content of allyl glycidyl ether increased from 0.2 to 1.0 mass% (based on the solid content of animal glue binder), and reached a maximum value of 2.30 MPa at an allyl glycidyl ether content of 1.5 mass%. This demonstrated that 1.5 mass% was the optimum modifier content. Further increase to 2.0 and 2.5 mass% allyl glycidyl ether caused a slight decrease in sample strength to 2.21 and 2.10 MPa, respectively (Figure 4). Because allyl glycidyl ether reacted with the active hydrogens of animal glue, the molecular mass of the binder gradually increased, resulting in improved strength as the allyl glycidyl ether content increased. Maximum strength was obtained when all active hydrogens in the binder were completely replaced; however, when excess allyl glycidyl ether was added, hydrolysis and self-polymerization of the modifier occurred, which caused a slight decrease in strength of the standard dog bone sand specimens.

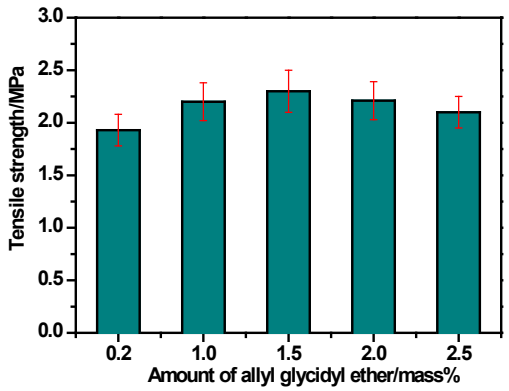

Figure 4. Effect of amount of allyl glycidyl ether on tensile strength of standard dog bone sand specimens

The effect of modification time on the viscosity of the animal glue binder was studied (Table 1). As the modification time increased, the viscosity of the binder decreased. This was attributed to hydrolysis of the glue. When the modification time was less than 1.5 h, the tensile strength of standard dog bone sand specimens increased as the modification time increased, reaching a maximum value of 2.58 MPa at a modification time of 1.5h. Further increase in modification time caused a decrease in the tensile strength of the standard dog bone sand specimens. Flowability of the standard dog bone sand specimens increased with the modification time due to decreased viscosity of the modified binder. When the modification time was short, the viscosity of the binder was too large and unsuitable for binding sand, so the tensile strength and flowability of the standard dog bone sand specimens were low. As the modification time increased, the animal glue was further hydrolyzed, accompanied by a decrease in viscosity: the modifier reacted with the glue, resulting in an increase in tensile strength and flowability of the standard dog bone sand specimens. An optimum tensile strength of 2.58 MPa was obtained at a modification time of 1.5h, when flowability of the samples was 1.95g. When the modification time exceeded 1.5h, the viscosity of the binder was too low, causing a decrease in sand strength.

Table 1. Effect of modification time on properties of modified animal glue binder and standard dog bone sand specimens

Modification time (h)	Intrinsic viscosity of modified animal glue binder ($\times 10^3$ mPa·s)	Tensile strength of standard dog bone sand specimes (MPa)	Flowability of standard dog bone sand specimens (g)
0.5	3.40±0.15	2.21±0.10	1.80±0.20
1.0	1.95±0.15	2.28±0.15	1.88±0.20
1.5	1.40±0.10	2.58±0.20	1.95±0.20
2.0	1.04±0.10	2.16±0.18	2.23±0.30
2.5	0.85±0.10	2.05±0.15	2.45±0.30

Elemental analyses of unmodified and modified animal binder are listed in Table 2. Compared with unmodified animal glue binder, the modified binder had slightly lower N and S contents and higher C and H contents due to the addition of allyl glycidyl ether.

Table 2. Elemental compositions of unmodified and modified animal glue binders

Animal glue	N (%)	C (%)	H (%)	S (%)	C/N ratio	C/H ratio
Unmodified animal glue	15.84	45.05	6.37	0.43	2.85	7.07
Allyl glycidyl ether modified animal glue	15.54	45.25	6.59	0.39	2.91	6.87

Figure 5 shows the FTIR spectra of unmodified animal glue binder, allyl glycidyl ether-modified animal glue binder, and allyl glycidyl ether. The appearance of an absorption band at 924 cm^{-1} and the increase in absorbance intensity at 1080 cm^{-1} for the modified binder illustrates

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that allyl glycidyl ether reacted with hydrolyzed animal glue [28].

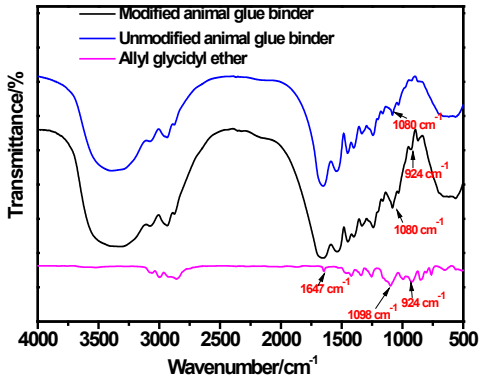

Figure 5. Fourier-transform infrared spectra of unmodified animal glue binder, allyl glycidyl ether-modified animal glue binder, and allyl glycidyl ether

Thermogravimetric curves of the modified and unmodified animal glue binders are presented in Figure 6. The mass loss below 200 °C was assigned to the removal of adsorbed water; that between 250 and 450 °C was attributed to the dehydration of macromolecular substances and loss of bound water in protein molecules. Compared with unmodified animal glue binder, the modified binder had a slightly lower mass loss below 300 °C, which indicated that it had slightly better stability.

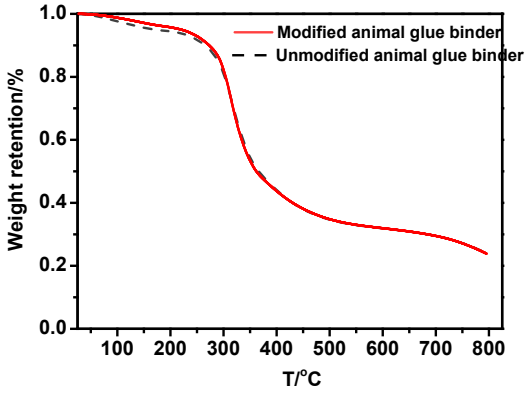

Figure 6. Thermogravimetric curves of unmodified and modified animal glue binders

Collapsibility of the mold has an important impact on the quality of a casting. Standard dog bone sand specimens that had been cured by blowing air were heated to 400, 500, and 600 °C for 5 min, cooled to room temperature, and then analyzed for residual tensile strength. As shown in Table 3, residual strength of the standard dog bone sand specimens decreased as the temperature increased: residual tensile strengths of standard dog bone sand specimens that were heat treated at 400, 500, and 600 °C for 5 min were 0.71, 0.10, and 0.00 MPa, respectively. The residual tensile strength of the sample heated at 500 °C for 5 min was close to 0.00 MPa. For comparison, for an animal glue binder modified with acrylic acid, ammonium persulfate, and glucose at a mass ratio of 30:3:15, residual tensile strengths of standard dog bone sand specimens heat treated at 500, 600, and 700 °C for 10 min were 0.39, 0.08, 0.00 MPa, respectively [21]. This indicated that the standard dog bone sand specimens using the allyl glycidyl ether-modified animal glue binder had good collapsibility. The mold could be easily removed on completion of casting and there was no residual grit on the device, thus producing a cast product with good quality.

Table 3. Residual tensile strength of standard dog bone sand specimens heat treated at different temperatures

Temperature (°C)	Residual tensile strength (MPa)[a]
400	0.71
500	0.10
600	0.00

Note: [a] Residual tensile strength was determined after cooling the standard dog bone sand specimens to room temperature.

The gas production of sand samples is an important indicator for the quality of casting. A lower value implies better quality because such samples have fewer defects and stomata. Figure 7 showed the gas evolution of sand samples with the allyl glycidyl ether-modified animal glue binder. Gas evolution was approximately 22.5 mL·g^{-1} at 850 ºC at 3600 s.

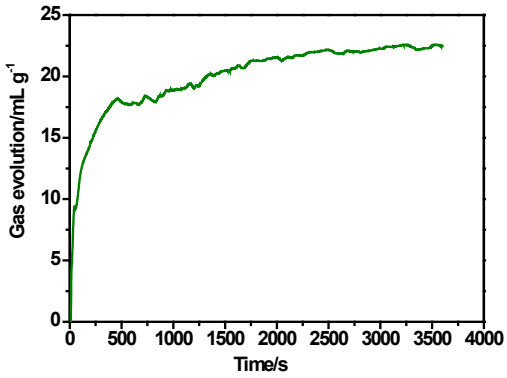

Figure 7. Gas evolution of sand sample with allyl glycidyl ether-modified animal glue binder

The water resistances of standard dog bone sand specimens were measured at 25 ºC and relative humidity of

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60%. As shown in Figure 8, samples prepared using the allyl glycidyl ether-modified animal glue binder had better water resistance than those using the unmodified binder. After 48 h, tensile strength of samples using the modified binder slightly reduced from an initial strength of 2.21 to 1.7 MPa; however, the corresponding tensile strength of samples using the unmodified binder significantly reduced from 1.83 to 0.56 MPa. The improved water resistance of samples using the modified binder was attributed to the interaction of allyl glycidyl ether with the active hydrogens of the animal glue [23].

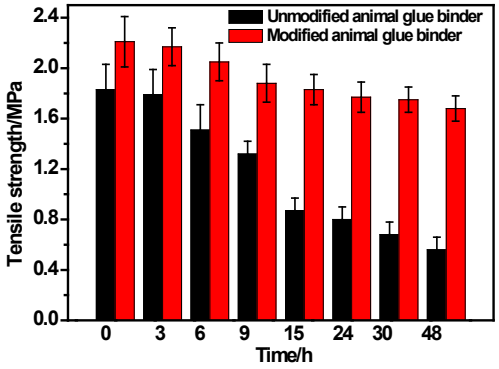

Figure 8. Water resistance of modified and unmodified animal glue binders

The bonding effect of the modified animal glue binder with quartz sand gravel is shown in the scanning electron micrographs in Figure 9. Native quartz sands had a smooth surface, and the gravels did not bind together (Figure 9a). After coating with the allyl glycidyl ether-modified animal glue binder, the surfaces of the sands were coated by the binder and became slightly rougher (Figure 9b). The grafted animal glue binder was uniformly dispersed in the gaps between the particles and bound them together, thus improving the sand strength.

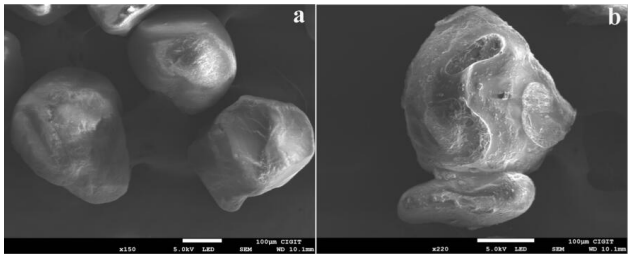

Figure 9. Scanning electron micrographs of (a) native quartz sand gravel and (b) quartz sand gravel coated with allyl glycidyl ether-modified animal glue binder

Conclusions

Modified animal glue binder was efficiently prepared using NaOH as the catalyst and allyl glycidyl ether as the modifier. The modification conditions were optimized using 4.0 mass% NaOH and 1.5 mass% modified animal glue binder, based on the solid content of animal glue binder, and a modification time of 1.5h. Using this modified animal glue as a casting binder only required a low-energy, environmentally friendly, and inexpensive curing procedure using compressed air at room temperature. The modified animal glue binder exhibited improved water resistance, good binding ability, low gas evolution, and appropriate collapsibility. The improved water resistance was ascribed the reaction of the modifier, allyl glycidyl ether, with the active hydrogen of the hydrolyzed animal glue. It can be used as a casting binder in the foundry industry.

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