Technical Note

High-Pressure Densification of Composite Lunar Cement

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Abstract: In order to minimize the binder content in composite lunar "cement," this paper uses JSC-1A lunar soil simulant and unsaturated polyester resin (UPR) to produce low-binder-content composite. Important system parameters such as compression pressure, compression duration, vibration, loading rate, loading number, and initial grain size distribution are analyzed. The most critical control variable is the compression pressure: at ∼350 MPa, the binder content is reduced to 6.5–8.7% by weight and the flexural strength is approximately 30–40 MPa. This research is important for in situ resource utilization for future lunar base construction and expansion. **DOI: 10.1061/(ASCE)MT.1943-5533.0002047.** © *2017 American Society of Civil Engineers.*

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Introduction

Very often composite materials are reinforced by fibers in order to maximize the stiffening and strengthening effects. Particulate fillers are also used, e.g., to enhance wear resistance or toughness, to improve formability, or to occupy space and reduce cost (Chawla 2013). Examples of particulate composites include carbon black–filled elastomers (Franta 2012) and concrete (Gani 1997). Usually, the binder (matrix) content in a particulate composite is in the range of 30–70% by weight (German 2016). In some special cases, e.g., in a polymer cement where the polymeric matrix is much more expensive than the sand/cement filler particles, the binder content may be lowered to ∼15% by weight (Chandra and Ohama 1997).

Space exploration missions continue to demand new composite materials that meet increasingly high requirements and tight constraints. Although cost is not a sensitive factor, the limitation on weight and size leads to unique challenges, e.g., the low binder content of composite lunar "cement" (CLC). A CLC is formed by bonding lunar soil grains with a small amount of polymer binder (Casanova and Aulesa 2000). It can be used for future construction and expansion of lunar bases and outposts, on-site fabrication of large-scale research and space facilities, and so on. Composite lunar cement is strong and its processing is simple and energy efficient. With the complexity of machinery and energy consumption taken into consideration, it is desirable that CLC can be based entirely on lunar regolith fines without the use of large aggregates.

Polymer was chosen as the binder in CLC because of its light weight, high strength, and broad working temperature range compared with other binder candidates (Ruess et al. 2004; Meyers and Toutanji 2007). Early studies in this area investigated polymer-clay nanocomposites (Qiao et al. 2007a, b). Over the last few years, other polymer binders have been investigated (Chen et al. 2008, 2015, 2016; Koh et al. 2010). One problem in these studies is the relatively high polymer content, often ranging from 15 to 20% by weight. In one work in which spherical silica beads were employed as an analogue to lunar soil grains, with an appropriate particle size gradation, the binder content was reduced to ∼8% by weight (Chen et al. 2015). However, when the inorganic particles were irregular-shaped JSC-1A lunar soil simulate (Chen et al. 2016), even with a precisely controlled grain size distribution the polymer content had to be higher than ∼15% by weight; otherwise the structural integrity of the CLC was poor. Regular processing techniques of composites, e.g., vigorous mixing or using plasticizers or spherical fillers, either fail to work when the binder content is below ∼15% by weight or are irrelevant to space construction. Important processing parameters, particularly the compression rate and pressure, compression duration and time, must be reinvestigated.

This paper investigated the key parameters of processing of particulate composites based on lunar soil simulant in order to decrease the binder content while maintaining a relatively high flexural strength. Compaction pressure was identified to be the most critical control variable.

Experiment

The inorganic filler was JSC-1A lunar soil simulant developed by the Johnson Space Center and provided by Orbitech (Madison, Wisconsin). It has similar composition, grain size, and grain shape as Apollo lunar soil samples, and has been widely employed as an analogue to lunar regolith in scientific research (Hill et al. 2007). The binder was an unsaturated polyester resin (UPR) (404 Tooling Polyester Resin—Isophthalic, US Composites, West Palm Beach, Florida), with methyl ethyl ketone proxide (MEKP; US Composites) as the initiator.

A cylindrical stainless steel load cell was produced with an inner diameter of 19.1 mm and a height of 50.8 mm. It contained two 19-mm-diameter stainless steel pistons with a length of 25.4 mm. The initiator and the UPR first were thoroughly mixed in a 50-ml beaker by a lab spatula for 1 min, with an initiator content of 1.5%. Approximately 15% by weight UPR–initiator mixture was added to ∼5 g JSC-1A grains in a second 50-ml beaker, rested for 60 s, and vigorously mixed at ∼60 rpm for 1 min. The mixture then was

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Table 1. Control Variables of the CLC Processing

Control variable	Description	Remarks
Compression pressure, P_{max}	30–700 MPa	As P_{max} rises, the binder content, C, decreases to 8.0% by weight
Duration of compression at P_{max}	1–300 min	As the compression duration increases, the binder content, C, decreases
Vigorous vibration	Vibrating the load cell at P_{max}	With vibration, the binder content, C, decreases to 6.5% by weight
Initial filler grain size	As-received or sieve-analyzed JSC-1A particles	Little influence on binder content
Compression rate	0.1–0.9 mm/min	Little influence on binder content
Number of compressions	Repeated compression for 1–4 times	Little influence on binder content

placed in the load cell and compressed by a type-5582 Instron machine (Instron, Norwood, Massachusetts) through the top and bottom pistons. The peak compaction pressure, P_{max}, ranged from 30 to 700 MPa. The compression rate ranged from 0.1 to 0.9 mm/min. After P_{max} was reached it was maintained for 1 min to 5 h, and the pressure was removed at the same rate as loading. For some samples, the compaction process was repeated multiple times. For some samples, before and/or during the compaction the load cell was vibrated by an Exen ELV8 piston vibrator (Tokyo, Japan) from the exterior. For some samples, prior to compaction the JSC-1A grain size distribution was controlled through sieve analysis using a W. S. Tyler (Mentor, Ohio) Ro-Tap 8-Inch Sieve Shaker with a 5-tray sieve stack; the mesh sizes, from the top to the bottom, were 500, 112, 90, 25, and 20 μm, respectively; the JSC-1A sample was formed using 90–112 μm grains and 20–25 μm grains, with a mass ratio of 765:235 (Chen et al. 2015). Table 1 summarizes the control variables.

The densified mixture of JSC-1A and UPR was cured at 100°C in air for 1 h in a VWR 1330GM (Radnor, Pennsylvania) oven and cut into beam samples by a MTI (Richmond, California) high-speed diamond saw (Fig. 1). The sample surfaces were thoroughly polished with 320-grit sandpaper.

Flexural strength, R, of the beam samples was measured in the type-5582 Instron machine, in accordance with ASTM C78/C78M-16. The two ends of a beam sample were supported by two smooth steel pins. At the middle point of the two support pins, the Instron machine compressed the sample at a speed of 3 mm/min. The flexural strength was calculated as $R = (3/2)(F_f L/bd^2)$ (Boresi and Schmidt 2003), where F_f is the failure force; $L \approx 19$ mm is the distance between the support bars; and $b \approx d \approx 6.3$ mm are, respectively, the sample width and height.

Because the pistons and the inner surface of load cell loosely fit with each other, during compaction a certain amount of binder was squeezed out. The final binder content was measured by a Perkin-Elmer (Waltham, Massachusetts) Thermalgravimetric Analysis (TGA) machine. Approximately 50 mg of CLC was harvested from the fracture surface of the tested beam sample, near the bottom edge where the final failure was initiated. The CLC powders were placed in the TGA machine and heated to 550°C for 1 h at a heating rate of 20°C/min. Fig. 2 shows a typical TGA curve. Fig. 3 shows the relationship between the final binder content, C, and the peak compaction pressure, P_{max}. Fig. 4 shows the relationship between the flexural strength, R, and the final binder content, C.

Discussion

In TGA measurement, the powder sample is heated to the set point and its mass variation is recorded continuously. For a reference sample of JSC-1A grains which contained no UPR binder, the mass loss typically was less than 0.1%, indicating that JSC-1A simulant is stable up to 550°C. The large mass loss of CLC at approximately 400°C shown in Fig. 2 should be related to the decomposition of UPR binder (Pascault et al. 2002). When the temperature exceeded ~500°C, the UPR decomposition was complete and the TGA curve became flat again. In the following discussion, the final binder content of compacted CLC, C, is taken as the mass loss in the TGA test.

The final binder content, C, was sensitive to the peak compaction pressure, P_{max} (Fig. 3). Before compaction, the JSC-1A grains were fully wet by ~15% by weight UPR, and the mixture was a slurry. Upon compression, JSC-1A grains were crushed and closely pressed together. The crushing strength of JSC-1A was approximately 20–30 MPa (Chen et al. 2016), lower than P_{max}. Although it is energetically favorable for the binder to wet the JSC-1A grain surfaces, the high pressure drove a considerable portion of UPR out of the mixture, which in turn flowed out of the steel load cell through

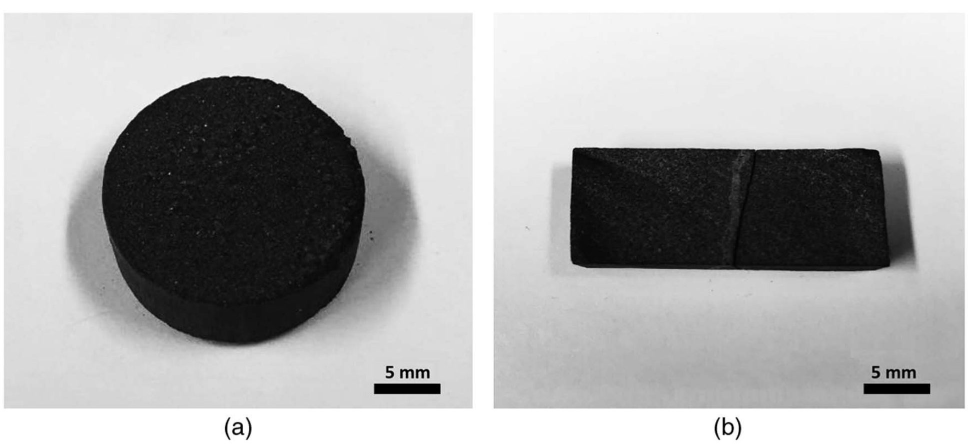

Fig. 1. (a) Disk-shaped CLC sample removed from the load cell after curing; (b) tested beam sample

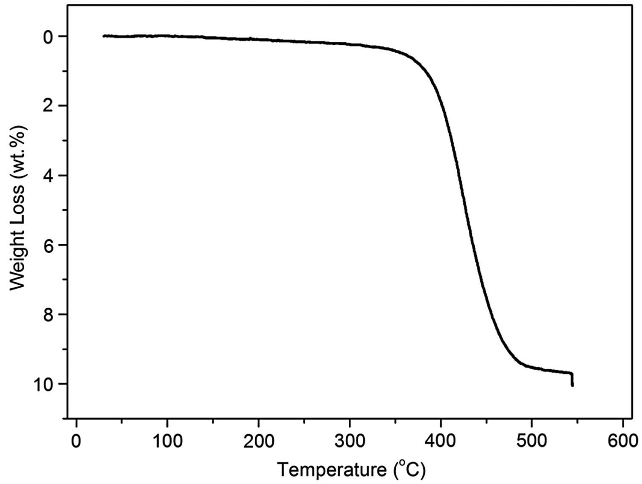

Fig. 2. Typical TGA curve; the mass loss around 400°C is associated with the decomposition of UPR binder; the mass loss of a reference sample without UPR was less than 0.1% by weight

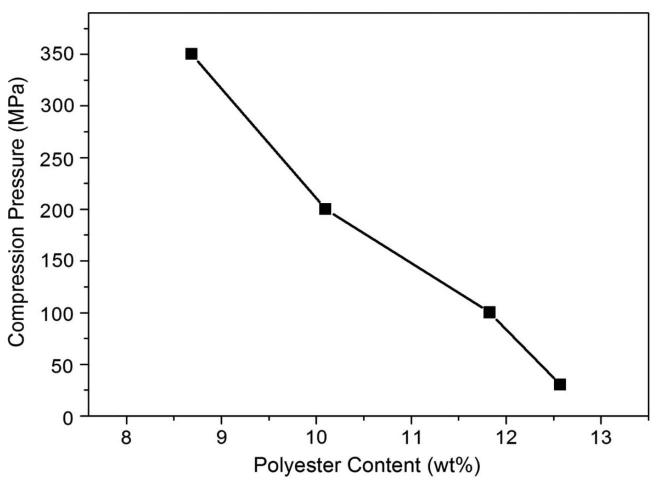

Fig. 3. Final binder content as a function of the peak compaction pressure, P_{max}; the compaction duration was 1 min; the compression rate was 0.3 mm/min

the piston gaps. As P_{max} increased, JSC-1A grain crushing and densification was promoted, reducing the free space between the grains. At P_{max} of 30 MPa, C was 12.6% by weight, comparable with the result of conventional composite processing (Chen et al. 2016). The final binder content linearly decreased by nearly 50% to ~8.7% by weight when P_{max} reached 350 MPa.

Fig. 4 shows the measured testing data of flexural strength (R). The variation in C did not influence R; R varied in the range from 30 to 40 MPa as C decreased from 12.6 to 8.6% by weight. This strength value was much higher than that of portland cement (3–5 MPa) and steel reinforced concrete (15–20 MPa) (Wight 2002), satisfactory for a large number of structural applications. The compaction-induced binder content reduction did not increase the defect density. The JSC-1A grains were well bonded. The final binder content was dominated by the volume fraction of the free space.

When the peak compaction pressure was further increased to 700 MPa, the final binder content was slightly reduced, from 8.6 to 8.0% by weight. The benefit was incremental, whereas the energy consumption and the requirements of load cell strength were much higher. The optimum P_{max} is approximately 350 MPa, above which the squeezing effect tends to saturate.

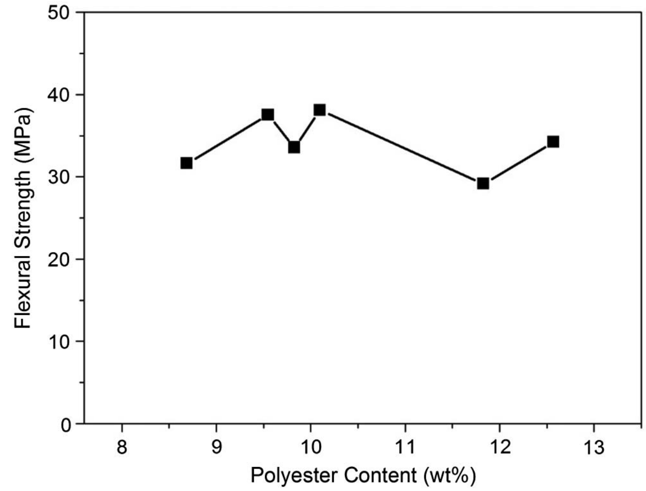

Fig. 4. Flexural strength as a function of the final binder content, C; the compaction duration was 1 min; the compression rate was 0.3 mm/min

When the duration of compaction increased from 1 min to 5 h, the final binder content decreased from 8.6 to 7.5% by weight. During the prolonged compaction, UPR underwent viscous flow. Most of the binder content reduction was achieved in the first minute; the subsequent slow reduction in C had a marginable benefit, but significantly decreased the processing rate and, especially when the compaction duration is longer than 1–2 h, may hurt the final strength.

Extensively vibrating the JSC-1A and UPR mixture at P_{max} from the exterior of load cell decreased C from 8.6 to 6.5% by weight; R remained at the same level of 30–40 MPa. The vibration operation does not demand extra processing time or complex equipment. It helps minimize the system free energy as the JSC-1A grains slightly rotate and shear.

The compression rate, the number of compression operations, and the initial JSC-1A grain size distribution had no evident influence on the final binder content and the flexural strength. Because of the relatively small JSC-1A grain size, the compression procedure was sufficiently slow to be considered as quasi-static and thus, in the ranges of compression rate and loading number under investigation, the system configuration was quasi-equilibrium. The initial JSC-1A grain size was a trivial factor because the grains were crushed and the actual grain size distribution in the compacted and cured CLC sample was determined by the compaction procedure.

Clearly, a number of other issues must be solved. For instance, the characterization procedure and materials improvement for ultraviolet (UV) resistance need to be investigated for both underground and aboveground CLC structures. If large lunar rocks are utilized as coarse aggregates or structural components, the amount of binder may be reduced much further.

Concluding Remarks

This paper investigated processing parameters of CLC based on JSC-1A lunar soil simulant and UPR. The goal was to achieve a high flexural strength and to minimize the binder content. High-pressure compaction had a pronounced beneficial effect. The optimum compaction pressure was approximately 350 MPa, above which the reduction in binder content was minor. Vibration

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considerably reduced the final binder content to 6.5% by weight. The flexural strength of the CLC was 30–40 MPa, insensitive to the final binder content. Most of the binder content reduction was achieved in the first minute of compression; prolonged compression did not result in a significant binder content reduction and may hurt the flexural strength. The influence of the compression rate, the number of loadings, and the initial grain size distribution was secondary.

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