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A COMPOSITIONAL STUDY OF REGOLITH COMPOSITES WITH CARBON NANOTUBE ADDITIVES FOR EXTRATERRESTRIAL CONSTRUCTION

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ABSTRACT

As extraterrestrial construction becomes an increasingly relevant goal in society, it is crucial to identify materials that balance high mechanical performance and ease of sourcing. One such candidate material, suitable for lunar habitat construction, is lunar regolith in combination with urea. This research aims to develop a novel lunar regolith composite for improved material strength. In this study we explore the relationship between the loading of carbon nanotubes within lunar regolith composites and their resulting modification of mechanical properties and porosity. Previous studies have shown the incorporation of carbon nanotubes in various applications such as fly ash composites, increases mechanical properties of interest for the material. The formulation of the composite material consists of lunar regolith, urea, distilled water, phosphoric acid, and carbon nanotube powder. The results of this study will include an assessment of the compressive strength for specimens containing carbon nanotubes at different weight fractions of 0%, 0.25%, 0.50% and 1.00%. Uniaxial compression tests demonstrate a maximum compressive strength of 5.82 MPa. We were able to achieve a 23% increase in compressive strength with carbon nanotube additives over composites containing no carbon nanotubes. However, space habitats require thorough and repeated testing to protect the lives at stake in these extreme environments. This increase in compressive strength allows the development of such materials and will allow for more freedom within the structural possibilities available in space architecture.

Keywords: Extraterrestrial Construction, Lunar Regolith Composites, Compressive Strength, Carbon Nanotubes.

1. INTRODUCTION

As space agencies work towards their goals of space exploration, their needs and interests in space architecture rapidly increase. To complete such architectural projects, there is a need for materials identification suitable for this purpose. Such materials that are being considered must meet qualifications for necessary material properties as well as fiscal feasibility. In relation to material properties, protection against radiation and thermal protection are crucial aspects for the materials applied in an extra-terrestrial environment context. Structural materials must also have a high performance in relation to mechanical properties, such as compressive strength and durability. The compressive strength requirements for lunar construction are 1/6 of the requirement on Earth, that is usually 35 MPa for a single floor [1]. Finally, the material must be resilient and reliable against extreme environments with large temperature changes and hold up under repeated cycling [2].

One candidate construction material that has recently come into investigation for applications in space architecture is regolith, which is the soil mixture that covers the surface of the Moon. Lunar regolith is composed of Al_2O_3, CaO, Na_2O, K_2O, light rare earth elements, and Th [3]. A primary advantage of utilizing regolith in the synthesis of building materials is the ability to locally source the material at the extraterrestrial location. This mitigates the extreme cost of transporting materials to space, with each pound of materials put into orbit costing approximately $10,000 [4]. Due to its accessibility, agencies including NASA (National Aeronautics and Space Administration), the ESA (European Space Agency), and ICON, among others, are looking to regolith. This material can be dug from lunar surfaces and combined with an alkaline solution or acidic solution to form a concrete-like composite material [1].

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This composite material could then be used as a main structural material for buildings or habitats.

Studies show that regolith-based materials can provide high levels of thermal protection and demonstrate efficiency in thermal insulation [5]. A previous study by Montes et al. looked into the mechanical and shielding properties of Lunamer, i.e., a regolith construction material, for prolonged lunar missions. The study found that the Lunamer formulation Luna-Iso-3d could reach up to 37.63 MPa and that an isostatically pressed Lunamer with a density of 2.29g/cm³ will improve the shielding properties [1]. For thermal properties, a study by Kim et al. found that cyclic temperature stress between −100 to 200 °C had little effect on sintered regolith samples coefficient of thermal expansion, displaying no visible fractures [6]. With these studies on regolith material showing high thermal resistance and ability to reach high compressive strength and shielding properties, regolith's properties allow the material to make a good candidate for lunar construction.

One composition of interest in regolith-based materials includes urea in the synthesis, which can potentially also be locally sourced in space from inhabitants' urine, sweat or tears. Pilehvara et al. found this mixture increases workability and has relatively high mechanical strength at 13 MPa with no freeze-thaw cycles, making a promising possible candidate material for space habitats. This study, sponsored by the ESA, found that urea mixed with regolith lowers the water percentage needed and delays initial and final setting times allowing better mixture flow during 3D printing [7]. Urea provides benefits for construction in space. Less water is necessary to create a sample that has high compressive strength and decreasing transportation needs. Urea is also beneficial for 3D printing due to the mixture having improved workability for the extrusion process.

Carbon nanotubes (CNTs) are materials that can be incorporated into existing material synthesis processes to improve environmental resistance [8-11] and the mechanical properties [12] of the material which is necessary for space architecture. For improved compressive strength, previous work by Chaipanich et al. showed that using 1% CNT in fly ash composites obtained the highest compressive strength of 51.8 MPa at 28 days [13]. Another study by Irshidat et al. disclosed that using 0.05% CNT creates an enhancement of the compressive strength by 15% compared to samples with an absence of CNT [14]. Based on these existing studies of carbon nanotube additives to cement-like mixtures, we predict the incorporation of CNT additives will improve the compressive strength of regolith composites.

In this study we explore the relationship between the loading of carbon nanotubes within lunar regolith composites and their resulting modification of mechanical properties and porosity. The formulation of the composite material consists of lunar regolith, urea, distilled water, phosphoric acid, and carbon nanotube powder. Specimens containing carbon nanotubes are synthesized at different weight fractions of 0%, 0.25%, 0.50% and 1.00%. The materials total 50g in weight and they are mixed together for homogeneity, then cast in a cylindrical mold and dried before curing. A methodology optimization is developed to increase performance through the minimization of porosity throughout the synthesis process. Uniaxial compression tests are used to analyze and compare the mechanical strength of the resulting materials. This result may be used to inform the practical application of these materials in space architecture as well as future studies on the optimization of composition for such materials.

2. METHODS

2.1 Synthesis

Work by Pilehvar et al. was used as a reference point in the initial attempts to synthesize high performance regolith composites [15]. From this basis, iterations on synthesis methods were made in the determination of a finalized methodology for our purposes as detailed in the following sections.

2.2 Materials

Composite materials within this work utilized for research and testing purposes are synthesized from lunar regolith, urea, distilled water, phosphoric acid, and carbon nanotube powder. Lunar regolith simulant is purchased from the company Exolith, where the chemical composition is given in Table 1. Urea granules, purchased from Lab Alley with a 99.0-100.5% assay (anhydrous basis), are used as a plasticizer which decreases the necessary total water. For the proper incorporation of these materials, both need to be further crushed in relation to their as purchased state.

Oxide	Wt%
SiO_2	51.2
TiO_2	0.6
Al_2O_3	26.6
FeO	2.7
MnO	0.1
MgO	1.6
CaO	12.8
NaO_2	2.9
K_2O	0.5
P_2O_6	0.1
LOI*	0.4
Total**	99.4

* Loss on Ignition

** Excluding volatiles and trace elements

TABLE 1: CHEMICAL COMPOSITION BY WEIGHT PERCENTAGE OF REGOLITH USED IN THIS STUDY.

An acidic solution is created using phosphoric acid and distilled water with a 32% composition of the regolith sample. Phosphoric acid is used as a pH modifier in a divergence from past work using an alkaline solution. During the synthesis reaction the donor hydrogen molecules bond with the regolith compound, thereby stabilizing the composite.

2.3 Porosity Mitigation

In a divergence from the comparable literature using sodium hydroxide [14, 16-17], this research uses phosphoric acid for preparing the acidic solution. Preliminary attempts to synthesize composites using sodium hydroxide were not successful due to its highly basic nature and causing a release of gas when mixed with regolith. Internal gas generation then caused pores to form throughout the material as it cured, lowering the compressive strength of the composite. This could be a result from the specific regolith simulant used. Phosphoric acid is used as a substitute within this work due to its less basic nature, causing a slower incorporation and preventing bubble formation.

Further issues with the porosity of the samples were observed to originate from non-uniformity in the mixture pre-cure, causing visible bubble formation in the mixture seen in Figure 1. One way this was overcome was by crushing the regolith, which originally had a particle size distribution of less than 1000 µm, to reach a 0.25 µm or less particle size distribution. This step was critical and also taken for the urea granules.

In addition to modifications on the mixture in the synthesis process, the methodology was also refined to mitigate porosity in the final material. A surface tension modifier was used on the inside of the mold to prevent trapped air from adhering to the walls of the mold. Before the curing process, the mixture is placed in a degassing chamber to further decrease porosity. Finally, when the mixture is in the mold, it is placed on a vibration machine to remove any other air trapped inside the specimens.

FIGURE 1: SAMPLES SHOW THE IMPACT BEFORE AND AFTER THESE CHANGES DESCRIBED IN THIS SECTION.BEFORE (LEFT) HAVING BUBBLY TOP AND VISIBLE PORES, AFTER (RIGHT) HAVNG SMOOTHER EDGES AND FLAT TOP.

2.4 Mixing, Casting & Curing

Preliminary tests were completed starting with reference points from the literature and iterated on to locate the most functional proportion of acidic solution to regolith. From there, different compositions of CNTs are tested with 0%, 0.25%, 0.50% and 1.00% weight percent of CNTs. The range of this loading was chosen to span the range of composite compositions possible, where going past the upper limit we observed complete structural failure after synthesis due to an increased dryness of the mixture. The composition of the tested composites is given in Table 2.

For composite specimen preparation, the regolith, phosphoric solution, urea, and CNT totaling 50g, were mixed together for 3 min to achieve a homogenous and uniform mixture. The mixture consistency that exhibited best on the compressive tests, was damp powder instead of a slurry. After degassing, the material is then packed into cylindrical molds at a size of 3 × 3 × 3 cm^3. Overfilling should be prevented to ensure testing uniformity. When the mixture is packed into the mold, the mold is placed on a vibration machine. After casting into the molds, the samples are left out in open air for 8 days. This setting time was chosen from testing a set of samples with a setting time of 24 hrs, 4 days and 8 days. The samples left for 8 days held up the best when demolding. The setting time allowed for the mixture to precure, then be demolded for improved even heating. After demolding, the samples are then placed in a heating chamber at a temperature of 80 °C for 8 h.

2.5 Testing methods

2.5.1 Compressive Test

The MTS universal mechanical testing system was used for this study. The compressive strength tests were performed on the four samples containing CNTs of 0%, 0.25%, 0.50% and 1.00%. The tests were controlled using a 0.017 mm/sec displacement rate. The load cell range used has a range of 4448.22 N.

2.5.2 Optical Microscopy

The composites were sectioned vertically, and a microscope was used to examine the four different composites. Two tests were captured, one looking at porosity percentage and other the quality of dispersion of the CNT.

CNT (g)	Wt%	Regolith (g)	Wt%	Urea(g)*	Wt%	Water(g)**	Wt%	Phosphoric Acid (g)**	Wt%
O	0.00	37.04	74.07	1.11	2.22	7.59	15.17	4.27	8.53
0.125	0.25	36.94	73.89	1.11	2.22	7.57	15.13	4.26	8.51
0.25	0.50	36.85	73.70	1.11	2.21	7.55	15.09	4.25	8.49
0.5	1.00	36.67	73.33	1.10	2.20	7.51	15.02	4.22	8.45

*Urea to Regolith Ratio: 3%
**Acidic Solution to Regolith Ratio: 32%
Total Weight: 50g

TABLE 2: MIXTURE COMPOSITION OF THE LUNAR REGOLITH SAMPLES BY GRAMS AND WEIGHT PERCENTAGE.

3. RESULTS AND DISCUSSION

3.1 Compressive Test

The compressive strength of the samples containing carbon nanotube additives at a weight percent of 0%, 0.25%, 0.50% and 1.00% are shown in Figure 2. In this testing group, we observe a positive relationship between composition of carbon nanotube additives and the compressive strength up to the 0.50% composition, whereafter strength decreases. The 0.50% composite achieves the highest strength measured compressive strength 5.82 MPa. This result is a 23% increase compared to the composite containing no CNT.

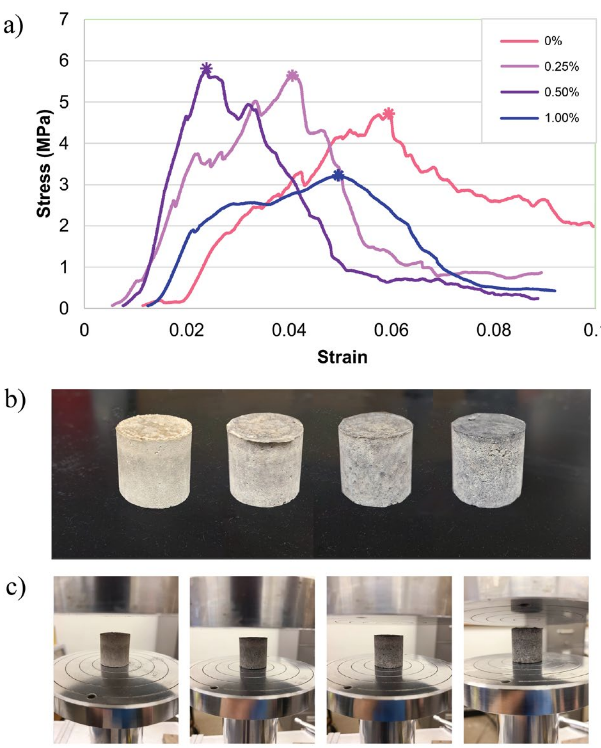

FIGURE 2: (a) COMPRESSIVE STRENGTH OF LUNAR COMPOSITES AT DIFFERENT CNT WEIGHT PERCENTAGES. STAR (*) INDICATE MAXIMUM STRENGTH (MPa) ACHIEVED. (b) SAMPLES TESTED, ORDER LEFT (0%) TO RIGHT (1.00%) INCREASING CNT WT%. (c) TESTING SET UP.

Analyzing the data trends of Figure 2a, the shift towards higher CNT compositions between 0%-0.50%, increases the ultimate compressive strength and decreases the strain at which it is achieved, corresponding to a more brittle behavior. Increasing the composition to 1.00% CNT caused the material to become too brittle and the trend of the line changes. This is consistent with preliminary testing where compositions above 1.00% experienced catastrophic failure after synthesis.

This study's samples are lower in strength compared to existing literature; the overall compressive strength of the samples achieved a maximum 13 MPa with 0 freeze-thaw cycles [7, 14]. This differentiation could be due to procedure, material differences, or specimen shape inconsistencies. Overall, the goal of the study is to focus on the CNT effects on the compressive strength rather than trying to achieve the highest compressive strength reported.

3.2 Optical Microscopy

Images gathered using optical microscopy are shown in Figure 3 examining the porosity of the different CNT concentrated samples. From these images, we observe a negative relationship between porosity and concentration of CNT, with the samples with the largest carbon nanotube percentage correlating to a lowest porosity as shown in Figure 5d. The samples are cleaved vertically, and the porosity is locally measured along the line of separation. The highest porosity locally measured was recorded in the samples that were taken. Samples with a 0%, 0.25%, 0.50% and 1.00% concentration of carbon nanotubes had a measured 11.32%, 10.4%, 8.134% and 5.715% porosity, respectively (ImageJ).

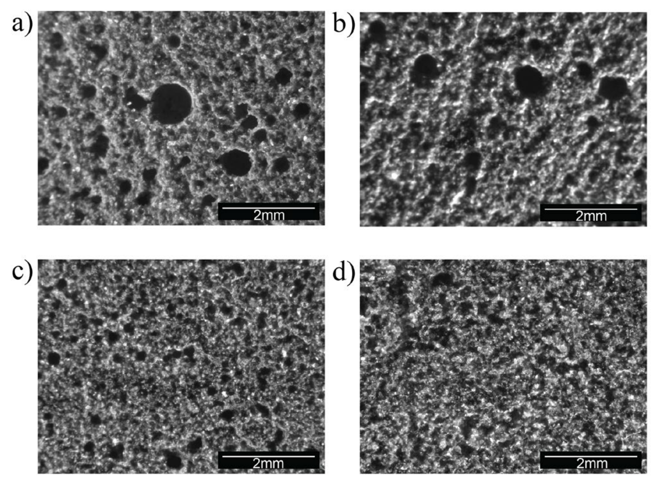

FIGURE 3: MICROSCOPIC IMAGERY OF THE SAMPLES VERTICALLY SECTIONED. (a) 0% CNT SHOWING LARGEST PORES. (b) 0.25% CNT. (c) 0.50% CNT. (d) 1.00% CNT SHOWING SMALLEST PORES WITH ROUGH TEXTURE.

The values of porosity measured in this work aligns with existing literature [18] with lower porosity observations being less than 0.50% and highest porosity being around 8%. This is also aligning with the standard values for concrete, with a porosity of 9-10% [19]. The relation observed between CNT and lower porosity can be also seen in a previous study by Nochaiya et al. that investigates the effect of CNT on Portland Cement [20]. Furthermore, our correlation of porosity and strength also is supported with existing literature showing that a higher percent of porosity lowers the compressive strength of the composite [18]. The microscopy images gathered shows an increase in brittleness as the CNT percentage increases. This is concluded from the observed roughness of the plane. This brittle characteristic of high CNT concentration samples also supports the compressive test observations detailed in the previous section. Further qualitative observations of the microscopic images in Figure 4 give insight into the characteristics of CNT dispersion for these composite materials. In most cases, the CNTs are visually observed to be well dispersed across the entire sample. However, as the CNT additives in the sample increase, the dispersion of these additives tends to decrease which leads to areas of high concentration. This could impact the compressive testing by having different areas in the composite being stronger or more brittle.

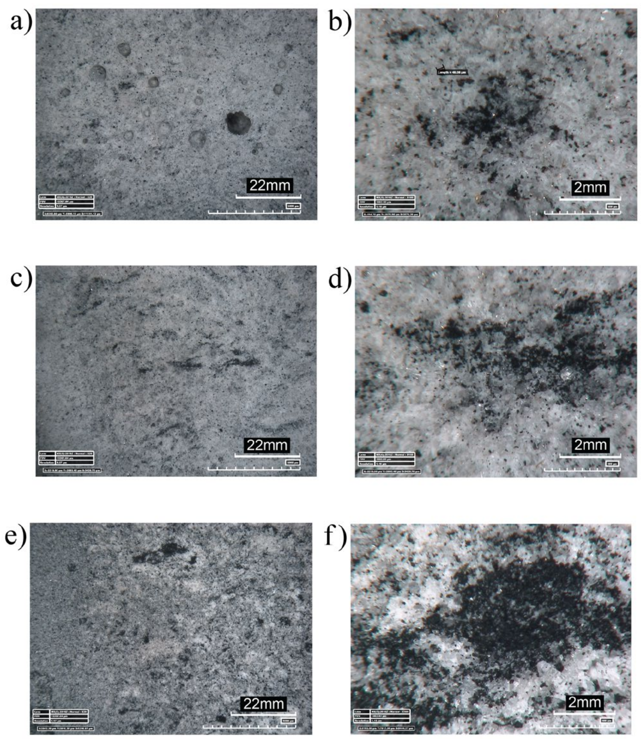

FIGURE 4: MICROSCOPIC IMAGERY OF SAMPLES CNT DISPERSION. (a), (b) 0.25% CNT. (c), (d) 0.50% CNT. (e), (f) 1.00% CNT. (a), (c), (e), IMAGERY OF INCREASING PROGRESSION OF CNT % IN SAMPLES. (b), (d), (f) CLOSE IMAGERY OF HIGH CONCENTRATION AREAS WITH 1.00% CNT DISPLAYING LARGEST CONCENTRATION.

4. CONCLUSION

In this study, the effects of different amounts of carbon nanotubes in a lunar regolith composite were examined through the mechanical properties and the qualitative and quantitative structure of the composite. The following conclusions can be drawn from this work:

1. Incorporating multiple porosity mitigation strategies including degassing, shaking, using phosphoric acid, and crushing components, greatly increases the stability and strength of the resulting composite material.
2. Carbon nanotubes can be used to increase the compressive strength of regolith composites. In this work, a weight percentage of 0.50% carbon nanotube additives performed the best, achieving a strength of 5.82 MPa on the compressive tests, out of a sample set containing 0%, 0.25%, 0.50% and 1.00% weight percent of carbon nanotubes. The 0.50% CNT achieved a 23% increase in strength compared to the sample with 0% CNT.
3. Porosity was shown to decrease with an increased concentration of carbon nanotubes, which can also be correlated to compressive strength.
4. The relationship of carbon nanotube additives to strength shows a positive correlation up to the maximum, whereafter the increased concentration of nanotubes causes the increased brittleness of the material to dominate the performance.

The observations in this work demonstrate the high potential of carbon nanotube - regolith composites for space architecture. Expanded methodologies, including, temperature cycling, environmental resistance testing, vacuum pressure and thermal performance are recommended in order to assess the effects of carbon nanotubes on lunar regolith composites under the severe lunar conditions.

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