Electronic Supplementary Material (ESI) for RSC Sustainability.

Supplementary file: One-Step Facile Process for Extraction of Cellulose from Rice Husk and their use for Mechanical Reinforcement of Dental Glass Ionomer Cement

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Abstract:

Cellulose is a widely available and renewable biopolymer that can be extracted from different natural sources such as plants, bacteria, and algae. Rice husk is of special interest as a source of cellulose, as it is abundant and has a high cellulose content. The extraction of cellulose from rice husk has traditionally been a multi-step procedure that is time-consuming and involves the use of chemicals. However, in this study, a one-step facile method was developed and optimized for the extraction of cellulose from rice husks. The process involved adjusting the ratio of sodium hypochlorite solution to the mass of solid rice husk and the progressive removal of non-cellulosic constituents was confirmed via Fourier transform infrared (FTIR), scanning electron microscopy (SEM), and Maule's test. The extracted cellulose batches were then incorporated into glass ionomer cement (GIC) for dental applications. Mechanical properties of the GIC have increased in G12 and G18 vs an observable decrease in G21 (p=0.02, and p=0.01, respectively), including increased compressive strength and stiffness. The addition of different cellulose batches has a significant effect on the incorporated GIC in terms of increasing its strength and elasticity as for G12 and G18. On the contrary, G21 has shown an insignificant change in terms of strength and elasticity in comparison to the control. In conclusion, extracted cellulose can be extracted from rice husks using a one-step facile method, and its incorporation into GIC can enhance its mechanical properties and improve its potential use for dental applications.

MesH Keywords: Cellulose, Hypochlorous acid, Glass ionomer cement (GIC), Compressive strength

Fig S1. Rice husk from Oryza sativa L., the primary rice planted in Sharkia governorate, Egypt.

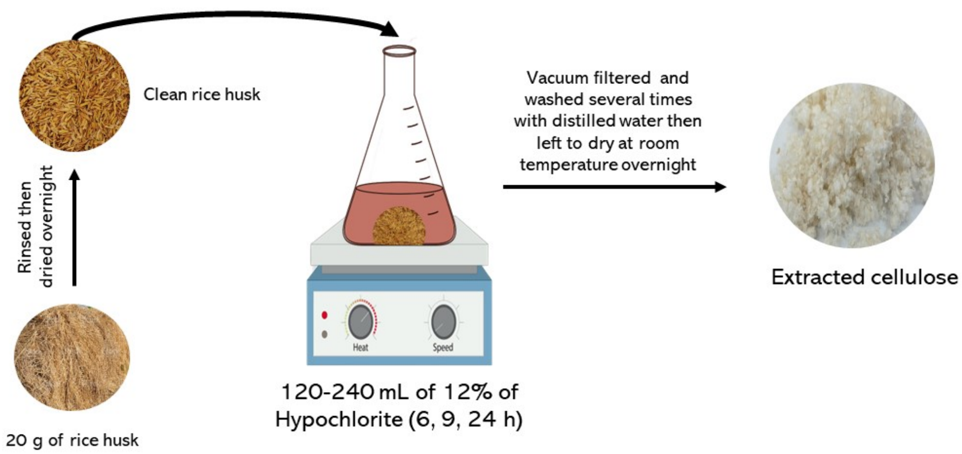

Fig S2. Preparation of extracted cellulose from rice husk using 12% sodium hypochlorite solution with different solid:liquid ratios and treatment time. The final product was vacuum filtered and washed with distilled water to obtain the extracted cellulose

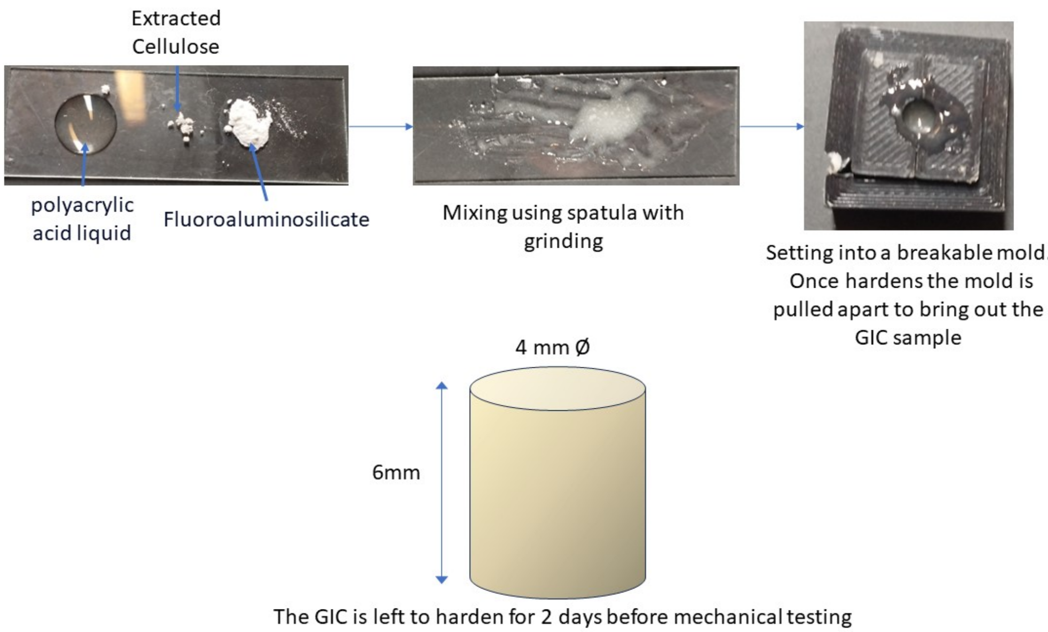

Fig S3. Fabrication steps of the GIC. Extracted cellulose and the fluoro-aluminosilicate powders were mixed using a vortex. Mixtures were placed within the mold and left for 5-10 min. The mold is removed and the obtained GIC cylinder was left to harden for 2 days before mechanical testing.

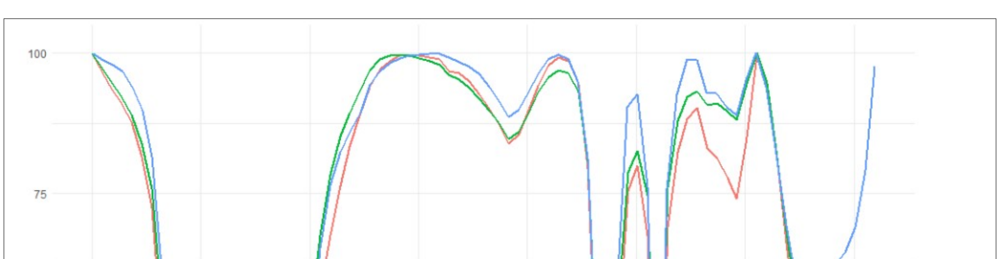

Figure S4. FTIR spectrum of C12 (S:L 1:12), C18 (S:L, 1:18), and C21 (S:L, 1:21) confirming the presence of cellulose.

Method

FTIR spectra of the obtained batches were recorded in transmittance mode accumulating 64 scans within the wavenumber range from 600 to 4000 cm^{-1}. The samples were mixed in smooth porcelain mortar and pestal with KBR in a mass ratio sample to KBR of 1:10.

Fourier-Transform infrared spectroscopic (FTIR) analysis:

FTIR analysis was used for confirming the successful cellulose extraction from the rice husk using sodium hypochlorite solution (**Figure S4**). Only three batches, C12, C18, and C21, which were incorporated into the GIC were selected for FTIR analysis. FTIR has confirmed the presence of cellulose in all tested samples due to the presence of peaks at 1640 cm^{-1} for C-O 6-membered lactone, 1400 cm^{-1} methylene groups, and 1165 cm^{-1}, 1115 cm^{-1}, and 1055cm^{-1} which represent the C-O stretching of alcoholic groups [1]. Although the SEM analysis and Maule's test have confirmed the presence of lignin in C12, no lignin-specific peaks were observed as they may have been masked by the cellulose peak patterns at 1700-1500 cm^{-1}, 1500-1000 cm^{-1}, and 700-500 cm^{-1} [1, 2].

Figure S5. Calibration curve of the dextrose using phenol-sulfuric acid.

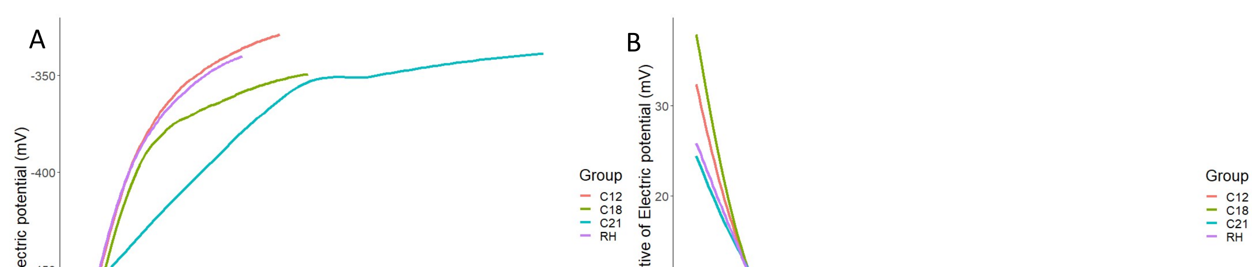

Figure S6. A) Titration curves for the determination of alpha-cellulose using ammonium ferrous sulfate as the titrant (volume mL). B) is the first derivative of the titration curve to identify the endpoint of the titration. C12, C18, and C21 are rice husk samples extracted using solid: liquid ratios of 1:12, 1:18, and 1:21 (rice husk to sodium hypochlorite). RH: rice husk.

**Mechanical compression data of GIC can be found in the attached pdf and Excel files.

References:

1. N. Atykyan, V. Revin and V. Shutova, *AMB Express*, 2020, **10**, 84.
2. S. Garoushi, J. He, J. Obradovic, P. Fardim, P. K. Vallittu and L. Lassila, *European journal of oral sciences*, 2020, **128**, 81-88.