SUPPLEMENTARY INFORMATION

StarCrete: a starch-based regolith biocomposite for extraterrestrial construction

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Experimental details

Materials

Martian Global Simulant (MGS-1) and Lunar Highlands Simulant (LHS-1) regolith powders were purchased from ExoLith Lab, with the precise composition of these simulants available at https://exolithsimulants.com. Potato starch (Whole Food Earth brand), corn starch (ARGO brand) and other starch sources were purchased from Amazon UK. Commercial grade $MgCl_2$, $FeSO_4$, acetic acid (24% v/v) and Na_2CO_3 were purchased from Amazon UK. Urea (laboratory grade, >99%) was purchased from Fischer Scientific UK. Freshly prepared human saliva was produced *in vivo* and incubated at body temperature before being carefully aliquoted into samples *via* oral transfer (*i.e.*, spitting). All reagents were used without further purification.

Initial StarCrete fabrication procedure

0.5 g of corn starch was mixed thoroughly with 12 g of MGS-1. 2 ml of deionised water was then added and further mixed into a homogenous paste. The mixture was then placed in a 50 ml glass jar (with aluminium cap, Amazon UK) and heated to 90 °C for 10 minutes, before being allowed to cool to room temperature (typically 12 – 16 °C) over about 60 minutes. Approximately 6 g of the resulting hybrid starch-regolith gel was then placed in a cylindrical steel container (inner diameter: 13 mm) and compressed with a well-fitting steel plug with a force of 3 metric tonnes (corresponding to 22.1 MPa of pressure). The cylindrical sample was removed and dried at 90 °C for 120 minutes. After drying, the sample was stored in an airtight bag prior to testing.

Optimised StarCrete fabrication procedure

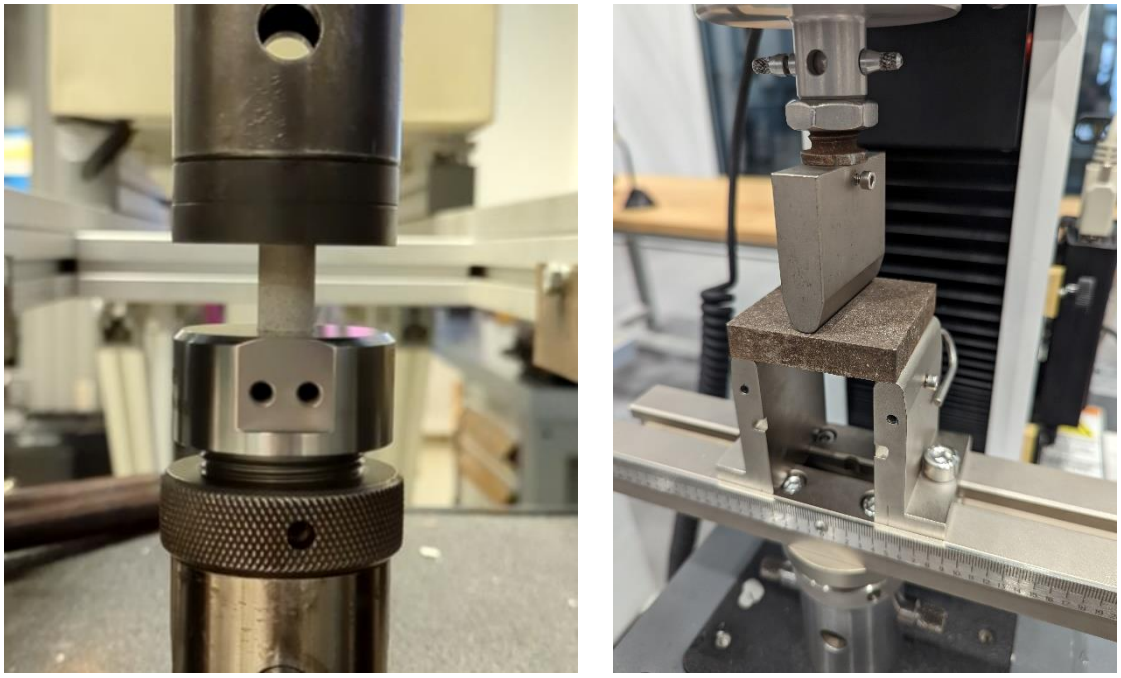
0.698 g of potato starch was mixed thoroughly with 12 g of MGS-1. 2.79 g of a 0.5 M $MgCl_2$ solution then added and further mixed into a homogenous paste. The mixture was then placed in a glass jar (with aluminium cap, Amazon UK) and heated to 120 °C for 120 minutes (*safety note: the glass vials sometimes explode due to excessive pressure*), before being allowed to cool to room temperature (typically 12 – 16 °C) over about 60 minutes. The hybrid regolith-starch gel was then dehydrated at 90 °C for 60 minutes, before being crushed into a powder with a pestle and mortar. 0.625 ml of deionized water was then added to rehydrate the powder, and mixed/crushed until homogenous. Approximately 6 g of this hybrid regolith-starch paste was then placed in a cylindrical steel container (inner diameter: 13 mm) and compressed with a well-fitting steel plug with a force of 3 metric tonnes (corresponding to 22.1 MPa of pressure). The cylindrical sample was removed and dried at 90 °C for 240 minutes. After drying, the sample was stored in an airtight bag prior to testing.

Uniaxial compression tests

To determine the UCS and compressive modulus of StarCrete sample specimens, we roughly followed the guidelines stated by ASTM International Active Standard C39/C39M-20 (Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens). Cylindrical specimens 13 mm in diameter and approximately 20 mm in length were subject to uniaxial compression tests using an Instron 5569 Series Universal Testing System (Instron Ltd., USA) that had been compliance corrected using a tungsten carbide disk. The tests were conducted using a 50 kN load cell at a rate of 1 mm min^{-1}. The tests were conducted at ambient temperature and humidity, with sample age ranging from about 24h to about two months.

Three-point flexural tests

To determine the flexural strength and modulus of StarCrete, tile-like specimens with dimensions of 55x55x12 mm were prepared and subject to three-point flexural tests using an Instron 3344 Series Universal Testing System (Instron Ltd., USA). Tests were conducted using a 2 kN load cell at a rate of 1 mm min^{-1}, with a sample span of 50 mm. The tests were conducted at ambient temperature and humidity, with sample age being about 5 days.



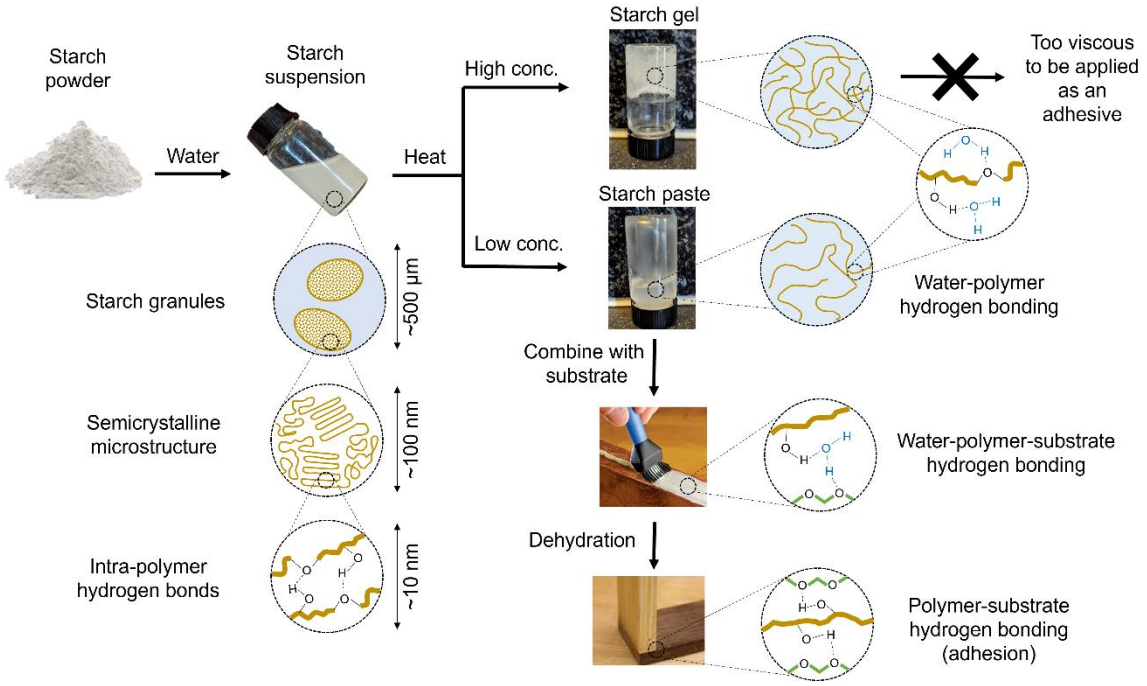
Camera images of the uniaxial compression test (left) and three-point bend test (right) setups.

Scanning electron microscopy (SEM) images

SEM images were taken using a Quanta 250 FE-SEM under secondary electron detection mode with an accelerating voltage of 8 kV and a working distance of 10 mm. Sample specimens were adhered to aluminium studs using conductive carbon tape and sputter-coated with an Au-Pd ally (approx. 10 nm in thickness) prior to imaging to enhance electrical conductivity.

Overview and mechanism behind starch-based adhesives and binders

To produce a typical starch-based adhesive or binder, starch powder is mixed with water and the resulting suspension is heated to between 50 – 85 °C. This hydrothermal treatment induces swelling and bursting of the semicrystalline starch granules, as hydrogen bonds are transferred from the metastable polysaccharide assemblies to water, releasing and solvating the largely linear amylose and branched amylopectin biopolymers.[1] This process is known as starch gelatinisation and results in a significant increase of the viscosity of the of the resulting hydrocolloid, with high concentrations resulting in robust hydrogels and lower concentrations resulting in viscous pastes – with the latter being suitable as an adhesive or binder (see figure below).[2] Bonding occurs after contact of the starch paste with the substrate and subsequent dehydration; here hydrogen bonds are transferred from solvated water-polysaccharide complexes to from intermolecular polymer-polymer crosslinks (cohesion) and polymer-substrate bonds (adhesion). This conventional method was adapted by Y. Kulshreshtha *et al.* to produce CoRncrete; here starch powder was first mixed with a sand substrate before addition of water and heating to induce gelatinisation. Since a viscous starch paste wasn't required as a processable intermediate, this allowed relatively high concentrations of starch to be employed – resulting in a robust hybrid polymer-inorganic gel matrix. On dehydration, this resulted in a composite material with a compressive strength as high as 26 MPa. G. Mansour *et al.* further developed the method, attaining compressive strengths as high as 30 MPa.[3] In this work, we built upon these findings and investigated the concept for extraterrestrial construction applications.



Scheme depicting the steps taken to produce a starch-based adhesive along with underlying molecular interactions

DoE experiment 1: 10-factor Definitive Screening Design (DSD)

In this experiment, JMP Pro 15 software (SAS Institute) was used to create a DSD – which is a screening design where main effects, second-order effects and quadratic effects are orthogonal (*i.e.*, are unbiased from one another).[4] The 10 input variables (factors) and their ranges – given in the table below – were inputted into the software, which outputted a table of 25 experimental runs. Run order was randomised to reduce the effect of any hidden variables biasing the results. Each run was conducted in triplicate, with the average value for compressive strength being input into the results table.

Table of factors and value ranges

Factor	**Classification**	**Value range**
Starch-regolith ratio (wt%)	Continuous	3 – 6
Effective starch concentration (wt%)	Continuous	20 – 25
Urea concentration (M)	Continuous	0 – 6
$MgCl_2$ concentration (M)	Continuous	0 – 1
Acetic acid concentration (vol%)	Continuous	0 – 5
Gelatinisation temperature (°C)	Continuous	70 – 90
Gelatinisation time (min)	Continuous	10 – 60
Compression force (Kgf x10^3)	Continuous	1 – 3
Drying temperature (°C)	Continuous	50 – 90
Drying time (min)	Continuous	120 – 240
Compression time (min)	Fixed	4
Resting temperature (°C)	Nuisance	12 - 16
Room humidity (%)	Nuisance	55 - 70

Table showing run combinations, run order, and resulting UCS

Run	**Starch-regolith ratio (wt%)**	**Effective starch conc. (wt%)**	**Urea conc. (M)**	**$MgCl_2$ conc. (M)**	**Acetic acid conc. (vol%)**	**Gel. temp. (°C)**	**Gel. time (min)**	**Comp. force (Kgf x10^3)**	**Drying temp. (°C)**	**Drying time (min)**	**UCS (MPa)**
1	6	25	6	0	2.5	90	10	3	50	120	0.80
2	4.5	25	6	1	5	90	60	3	90	240	35.00
3	4.5	22.5	3	0.5	2.5	80	35	2	70	180	20.42
4	6	20	0	1	0	90	60	3	50	120	20.52
5	3	20	0	0	5	80	10	3	90	120	5.35
6	3	25	6	0	0	70	60	3	70	120	14.34
7	6	20	6	1	5	70	35	3	90	120	6.79
8	3	25	0	1	0	90	10	3	90	180	2.12
9	6	20	0	1	5	90	10	1	70	240	18.94
10	6	22.5	0	0	0	70	60	3	90	240	19.95
11	6	25	0	0.5	5	70	10	3	50	240	0.26
12	3	20	6	1	0	70	10	3	50	240	4.72
13	3	25	0	1	5	70	60	2	50	120	0.99
14	3	22.5	6	1	5	90	10	1	50	120	5.83
15	3	20	6	0.5	0	90	60	1	90	120	22.50
16	6	20	6	0	0	90	10	2	90	240	7.40
17	6	20	6	0	5	70	60	1	50	180	0.48
18	3	20	0	1	2.5	70	60	1	90	240	0.72

19	6	25	6	1	0	80	60	1	50	240	1.25
20	3	25	6	0	5	70	10	1	90	240	7.06
21	4.5	20	0	0	0	70	10	1	50	120	0.32
22	3	25	0	0	0	90	35	1	50	240	1.05
23	6	25	0	0	5	90	60	1	90	120	31.42
24	3	20	3	0	5	90	60	3	50	240	15.38
25	6	25	3	1	0	70	10	1	90	120	3.50

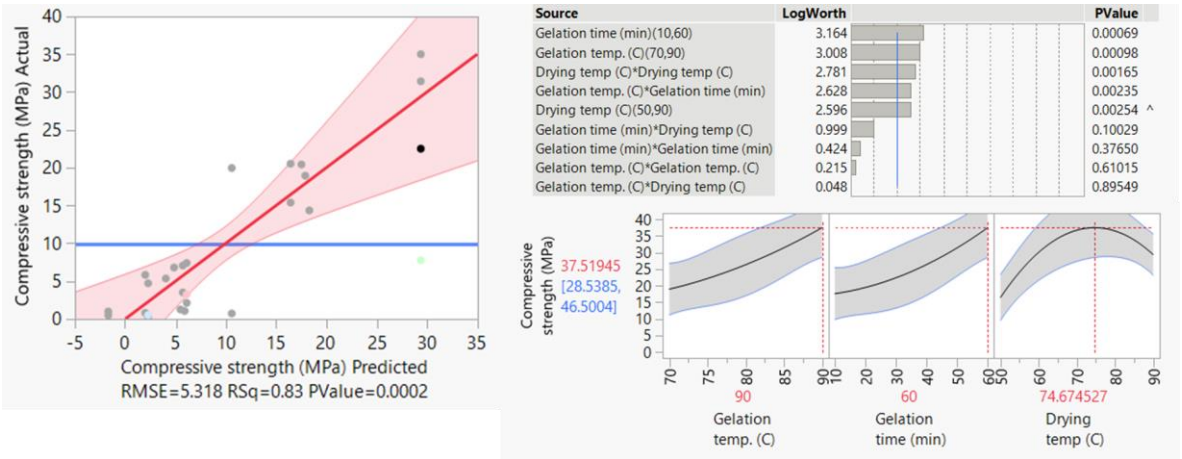
After conducting the experimental runs and inputting the UCS data into the results table, the Standard Least Squares method with an emphasis on Effect Screening was employed to construct a Response Surface Model (RSM) from the construct model effects. The combined model parameter estimates, presented in the table below, were used to produce the model without any further changes.

Table showing the combined model parameter estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	17.513	4.317	4.0568	0.0010*
Gelation temp. (C)	4.6283	1.1338	4.0821	0.0010*
Gelation time (min)	4.8298	1.1338	4.2598	0.0007*
Drying temp (C)	4.1006	1.1338	3.6167	0.0025*
Gelation temp. (C)*Gelation time (min)	4.6046	1.2604	3.6532	0.0024*
Gelation temp. (C)*Drying temp (C)	0.1684	1.2604	0.1336	0.8955
Gelation time (min)*Drying temp (C)	2.2075	1.2604	1.7514	0.1003
Gelation temp. (C)*Gelation temp. (C)	1.886	3.6218	0.5207	0.6102
Gelation time (min)*Gelation time (min)	3.3009	3.6218	0.9114	0.3765
Drying temp (C)*Drying temp (C)	-13.85	3.6218	-3.825	0.0017*

Statistic	Value
RMSE	5.318
DF	15

The results found that gelatenisation temperature, gelatenisation time and drying temperature were highly significant – and were likely eclipsing the effects of the other parameters. Using JMP software's prediction profiler and the "maximise desirability" function, it was suggested that higher gelatenisation temperatures and gelatenisation times would further increase the UCS of the resulting materials, as would having a drying temperature of 75 °C. Interpreting this data, it was likely that the starch had not fully gelatinised at the lower oven temperatures and times – meaning only the conditions in which high temperatures and times had been employed demonstrated good mechanical properties.



Details of resulting response surface model (RSM) and its predictions

DoE experiment 2: 2-factor on-face Central Composite Design (CCD)

The preceding DSD experiment predicted that the UCS of the materials could be improved by increasing the gelatinisation time and gelatinisation temperature (*i.e.*, the materials were currently "undercooked"). To find more optimal conditions, the experimental combinations from "run 2" of the preceding DSD experiment were all fixed aside from gelation time and temperature – which were increased. Run 2 was selected since these gave the highest UCS of all other combinations (35 MPa).

To conduct this experiment, JMP Pro 15 software was again employed to construct a classical CCD with 1 centre point and an axial value of 1 (*i.e.*, on-face). Note that this design is identical to a 2-factor 3-level full factorial experiment. The gelatinisation temperature and gelatinisation time were varied between 90 – 120 °C and 60 – 120 min, respectively. Initially, a drying temperature of 75 °C was employed as the preceding DSD indicated that this would be optimal, however the UCS values of the produced materials were lower than for when a drying temperature of 90 °C was employed. This suggested that a more complex multi-factor interaction was likely occurring, which was not detected by the DSD. To simplify ongoing experiments, drying temperature and drying time were fixed at 90 °C and 240 mins, respectively. This significantly increased the rate at which we could conduct experiments due to the drying oven being a rate-limiting step. Samples were also produced in duplicate, rather than triplicate, for this and ongoing experiments – since the initial DSD experiment found lower variability between sample specimens than expected. This also increased the rate at which we could conduct subsequent experiments.

A table of the factors and factor ranges, and a map of the explored parameter space, is given below.

Table of factors and factor ranges

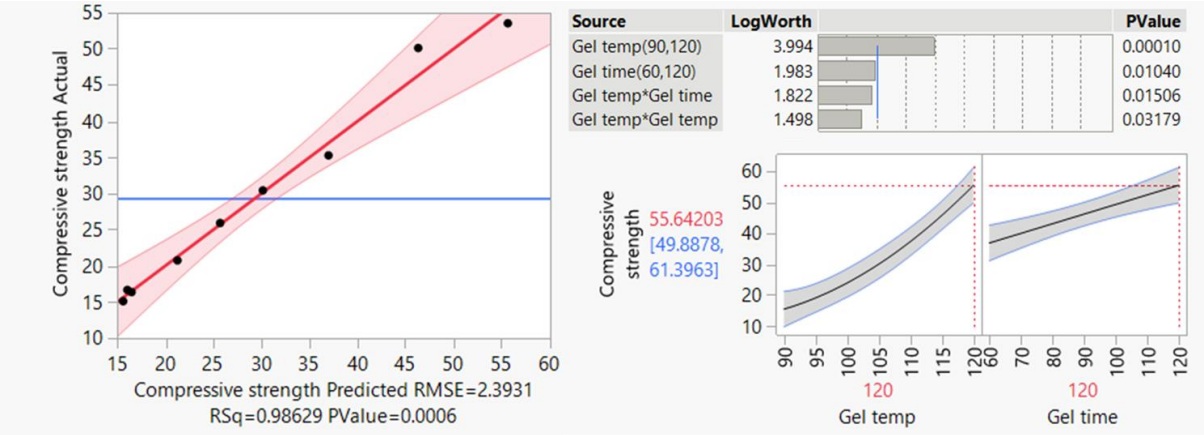
Factor	**Classification**	**Value range**
Starch-regolith ratio (wt%)	Fixed	4.5
Effective starch concentration (wt%)	Fixed	25
Urea concentration (M)	Fixed	6
$MgCl_2$ concentration (M)	Fixed	1
Acetic acid concentration (vol%)	Fixed	5
Gelatinisation temperature (°C)	Continuous	90 – 120
Gelatinisation time (min)	Continuous	60 – 120
Compression force (Kgf $x10^3$)	Fixed	3
Drying temperature (°C)	Fixed	90
Drying time (min)	Fixed	240
Compression time (min)	Fixed	4
Resting temperature (°C)	Nuisance	12 - 16
Room humidity (%)	Nuisance	55 - 70

A table showing the experimental runs, parameter combinations and corresponding patterns is given below. Note that run order was *not* randomised in this instance, and instead performed in batches based on oven temperature - this allowed the experiment to be conducted much faster than if run order was fully randomised.

Table showing run combinations, run order, and resulting UCS

Run	Pattern	Gel temp (°C)	Gel. time (min)	UCS (MPa)
1	−−	90	60	16.37
2	−+	90	120	15.10
3	+−	120	60	35.27
4	++	120	120	53.54
5	a0	90	90	16.66
6	A0	120	90	50.12
7	0a	105	60	20.75
8	0A	105	120	30.43
9	0	105	90	25.92

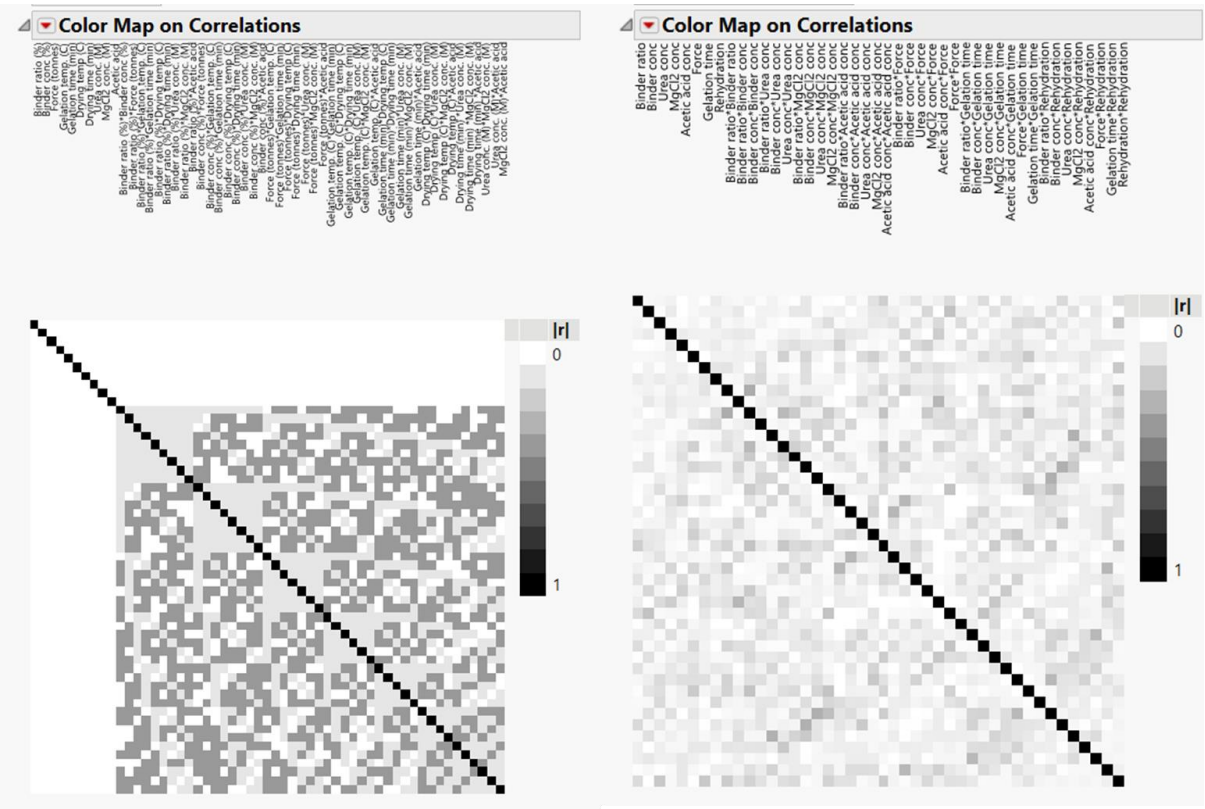
After inputting the UCS data into the results table, an RSM was constructed again using the Standard Least Squares personality with an Effect Screening emphasis. This produced a well-fitting model where there was a strong correlation between predicted and actual values for UCS (see figure below). Furthermore, the model strongly indicated that even higher gelatenisation temperatures and gelatenisation times would further improve UCS – however, urea undergoes thermal decomposition at temperatures above 150 °C to produce the poisonous gas isocyanic acid, so the temperature was fixed at 120 °C for the subsequent experiment as getting poisoned wasn't a desirable outcome.



Details of resulting response surface model (RSM) and its predictions

DoE experiment 3: 8-factor Custom Design

Having determined that a gelation temperature of 120°C was a good compromise between obtaining a high UCS and not producing a poisonous gas, this factor was fixed at that value. Sufficient process knowledge had now been obtained that a more in-depth DoE design was conducted, that would produce a higher-resolution RSM than the initial DSD. To depict this, a heat map on correlations for both the initial DSD and present custom design is given blow. Although the DSD had no confounding main effects and second-order effects by design, more complex interactions have quite substantial aliasing – as can be seen by the darker areas on the correlation map. Conversely, the custom design has a much lower degree of aliasing over the broader information space – and should produce a more reliable model given the degree of complex interactions likely occurring in this system.



Correlation heat maps displaying intensity of aliasing between factors for the initial 10-factor DSD (left) and the 8-factor Custom design (right)

To construct this experimental design, JMP Pro 15 software was again employed using the "custom design" feature. The identified parameters and parameter ranges, presented in the table below, were inputted into the software – and a RSM was specified as a required model output. Although 52 experimental runs were suggested, 54 runs were specified to allow the blocking of the experiment into 9 equal blocks of 6 (*i.e.*, 9 oven batches of 6 were produced – with the batch number being incorporated into the model to determine any significant batch-to-batch variability). Run order was again randomised to prevent nuisance variables from biasing the results.

Table of factors and factor ranges

Factor	**Classification**	**Value range**
Starch-regolith ratio (wt%)	Continuous	3.5 – 5.5
Effective starch concentration (wt%)	Continuous	20 – 25
Urea concentration (M)	Continuous	0 – 6
$MgCl_2$ concentration (M)	Continuous	0 – 1
Acetic acid concentration (vol%)	Continuous	0 – 5
Gelatinisation temperature (°C)	Fixed	120
Gelatinisation time (min)	Continuous	60 – 120
Compression force (Kgf $x10^3$)	Continuous	1 – 3
Drying temperature (°C)	Fixed	90
Drying time (min)	Fixed	240
Rehydration (%)	Continuous	5 – 7
Compression time (min)	Fixed	4
Resting temperature (°C)	Nuisance	12 - 16
Room humidity (%)	Nuisance	55 - 70

The specific factor combinations and run order is presented in the table below, along with the measured UCS data based on duplicate measurements. Notably, the 54^{th} run had an exceptionally high UCS of 71.1 MPa.

Table showing run combinations, run order, and resulting UCS

Run	**Block**	**Starch-regolith ratio (wt%)**	**Effective starch conc. (wt%)**	**Urea conc. (M)**	**$MgCl_2$ conc. (M)**	**Acetic acid conc. (vol%)**	**Gel. time (min)**	**Rehydration (%)**	**Comp. force (Kgf $x10^3$)**	**UCS (MPa)**
1	1	3.5	20	6	1	5	90	6	2	15.45
2	1	3.5	25	3	0.5	5	120	6	1	40.27
3	1	4.5	25	6	0.5	0	90	7	2	41.12
4	1	4.5	25	0	1	2.5	60	5	3	31.23
5	1	3.5	22.5	6	0.5	5	60	5	3	48.27
6	1	5.5	25	6	0	5	60	6	2	12.85
7	2	5.5	22.5	3	1	2.5	90	5	2	19.54
8	2	3.5	25	6	1	2.5	90	5	1	45.94
9	2	5.5	20	6	0.5	5	90	5	1	8.30
10	2	5.5	25	0	0	0	60	7	3	21.56
11	2	4.5	22.5	0	1	5	120	6	3	49.40
12	2	5.5	22.5	6	1	2.5	60	7	1	6.70

13	3	3.5	20	0	1	5	120	7	1	24.23
14	3	4.5	22.5	3	0.5	2.5	90	6	2	58.52
15	3	4.5	25	3	0	2.5	120	5	3	44.80
16	3	5.5	25	4.74	1	0	120	6	3	26.73
17	3	4.5	25	6	1	5	120	5	2	35.58
18	3	4.5	22.5	0	0	0	90	5	1	22.28
19	4	3.5	22.5	0	0	0	120	7	2	16.75
20	4	3.5	25	0	0	0	60	7	1	7.30
21	4	5.5	20	3	1	5	60	7	3	11.54
22	4	5.5	20	0	0	5	60	5	3	54.07
23	4	5.5	25	6	0	0	120	6	1	16.96
24	4	4.5	22.5	3	0.5	2.5	90	6	2	51.01
25	5	3.5	22.5	3	0.5	0	60	6	2	45.68
26	5	4.5	20	6	0	5	120	6	3	24.28
27	5	3.5	25	6	0	0	60	5	2	44.82
28	5	3.5	22.5	6	0	2.5	120	5	1	40.17
29	5	3.5	25	0	1	0	120	5	2	29.85
30	5	3.5	20	6	0.5	0	90	7	1	34.02
31	6	3.5	25	6	0.5	5	120	7	3	42.80
32	6	5.5	20	0	0	2.5	120	6	1	25.48
33	6	3.5	20	3	0.5	5	120	5	2	55.57
34	6	5.5	20	6	0	0	60	7	2	8.80
35	6	3.5	20	6	1	0	120	5	3	54.43
36	6	4.5	22.5	3	1	0	120	7	3	40.61
37	7	3.5	20	0	1	5	60	5	1	39.77
38	7	4.5	20	6	1	2.5	120	6	1	22.96
39	7	4.5	20	6	1	0	60	5	1	35.76
40	7	5.5	22.5	6	0.5	2.5	90	6	3	10.60
41	7	4.5	22.5	0	0.5	5	60	7	2	22.03
42	7	4.5	22.5	3	0.5	2.5	90	6	2	58.97
43	8	3.5	20	3	0	2.5	60	7	3	42.57
44	8	5.5	22.5	3	0	5	120	7	3	53.94
45	8	5.5	25	0	1	5	120	5	1	60.16
46	8	3.5	25	3	1	5	60	7	2	32.18
47	8	5.5	25	3	0.5	2.5	60	5	1	41.89
48	8	3.5	20	0	1	0	60	7	3	24.39
49	9	5.5	20	0	1	0	90	7	1	56.48
50	9	3.5	20	3	0	0	90	5	3	40.42
51	9	4.5	22.5	3	0	5	90	7	1	55.75
52	9	3.5	25	0	0	5	90	5	2	12.71
53	9	5.5	25	0	0.5	2.5	120	7	2	35.06
54	9	5.5	20	0	0.5	0	120	5	3	71.10

An RSM model was again constructed using the default Standard Least Squares personality, with an emphasis on Effect Screening using the REML method with unbound variance components. Since the produced model had a large number of effects with a low contribution

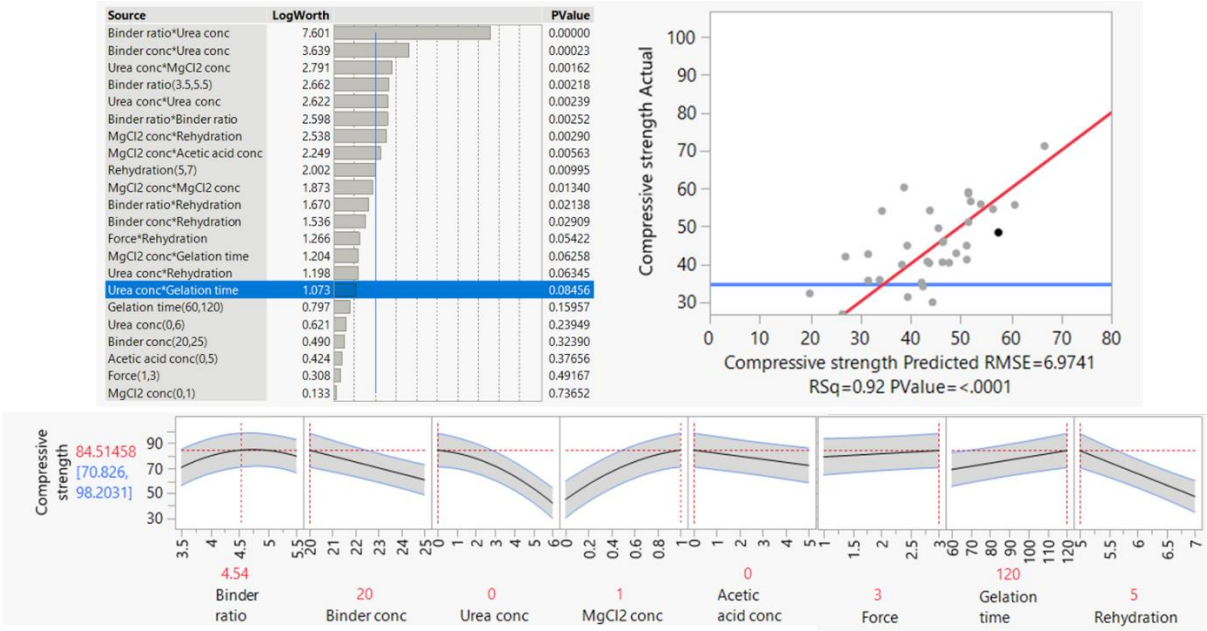
towards the final model – these were sequentially removed in order of significance (starting from least significant), until the LogWorth value for the least significant second order interaction (*i.e.*, urea conc.*gel. time, highlighted below) was above a value of 1. A table of the parameter estimates of the resulting model is presented below.

Table showing the combined model parameter estimates

Parameter Estimates

Term	Estimate	Std Error	DFDen	t Ratio	Prob>|t|
Intercept	51.480703	3.826939	18.12	13.45	<.0001*
Binder ratio(3.5,5.5)	-4.981443	1.477416	28.3	-3.37	0.0022*
Binder conc(20,25)	-1.358027	1.348206	23.87	-1.01	0.3239
Urea conc(0,6)	-1.655282	1.373406	24.79	-1.21	0.2395
MgCl2 conc(0,1)	0.4544379	1.335719	25.13	0.34	0.7365
Acetic acid conc(0,5)	-1.135941	1.260579	23.77	-0.90	0.3766
Force(1,3)	-0.915471	1.309368	22.3	-0.70	0.4917
Gelation time(60,120)	1.9960383	1.377263	25.23	1.45	0.1596
Rehydration(5,7)	-3.442277	1.224219	22.79	-2.81	0.0099*
Binder ratio*Binder ratio	-9.228496	2.771701	27.03	-3.33	0.0025*
Binder ratio*Urea conc	-13.57299	1.787272	28.55	-7.59	<.0001*
Binder conc*Urea conc	6.761403	1.574304	25.14	4.29	0.0002*
Urea conc*Urea conc	-8.41893	2.480909	24.09	-3.39	0.0024*
Urea conc*MgCl2 conc	-6.62517	1.907089	29.27	-3.47	0.0016*
MgCl2 conc*MgCl2 conc	-6.785423	2.536599	23.42	-2.68	0.0134*
MgCl2 conc*Acetic acid conc	-4.972254	1.653702	27.16	-3.01	0.0056*
Urea conc*Gelation time	-2.740993	1.520002	22.82	-1.80	0.0846
MgCl2 conc*Gelation time	2.8083915	1.43396	22.69	1.96	0.0626
Binder ratio*Rehydration	4.0627256	1.660543	26.39	2.45	0.0214*
Binder conc*Rehydration	3.7490934	1.612758	23.46	2.32	0.0291*
Urea conc*Rehydration	2.9028646	1.492496	24.27	1.94	0.0635
MgCl2 conc*Rehydration	-4.960623	1.48756	22.85	-3.33	0.0029*
Force*Rehydration	-3.424524	1.698022	25.85	-2.02	0.0542

The resulting model revealed some significant insights into the experimental system, as discussed in the main manuscript. The most significant insight being that acetic acid and urea had a detrimental effect on UCS and could therefore be dropped from the formulation – simplifying the process going forward.



Details of resulting response surface model (RSM) and its predictions

DoE experiment 4: 4-factor Custom Design

The insights gathered from the preceding experiment were subject to a final round of optimisation through another DoE Custom Design. Here, urea and acetic acid concentration were reduced to 0 as their inclusion had been found to be detrimental to UCS. The absence of urea meant higher gelation temperatures could be explored without the risk of producing isocyanic acid, so gelation temperatures up to 180 °C were investigated. Gelation time was also increased to 180 min as the previous experiment had indicated that longer times to be beneficial. Lower effective starch concentrations and higher $MgCl_2$ concentrations were also investigated based on predictions from the previous experiment, with all other factors being fixed at their most desirable levels (see table below). The variables were again input into JMP Pro 15 software using the "custom design" feature, which output 21 experimental runs of various factor combinations to complete.

Table of factors and factor ranges

Factor	**Classification**	**Value range**
Starch-regolith ratio (wt%)	Fixed	4.7
Effective starch concentration (wt%)	Continuous	15 – 20
Urea concentration (M)	Fixed	0
$MgCl_2$ concentration (M)	Continuous	1 - 2
Acetic acid concentration (vol%)	Fixed	0
Gelatinisation temperature (°C)	Continuous	120 – 180
Gelatinisation time (min)	Continuous	120 – 180
Compression force (Kgf $x10^3$)	Fixed	3
Drying temperature (°C)	Fixed	90
Drying time (min)	Fixed	240
Rehydration (%)	Fixed	5
Compression time (min)	Fixed	4
Resting temperature (°C)	Nuisance	12 - 16
Room humidity (%)	Nuisance	55 - 70

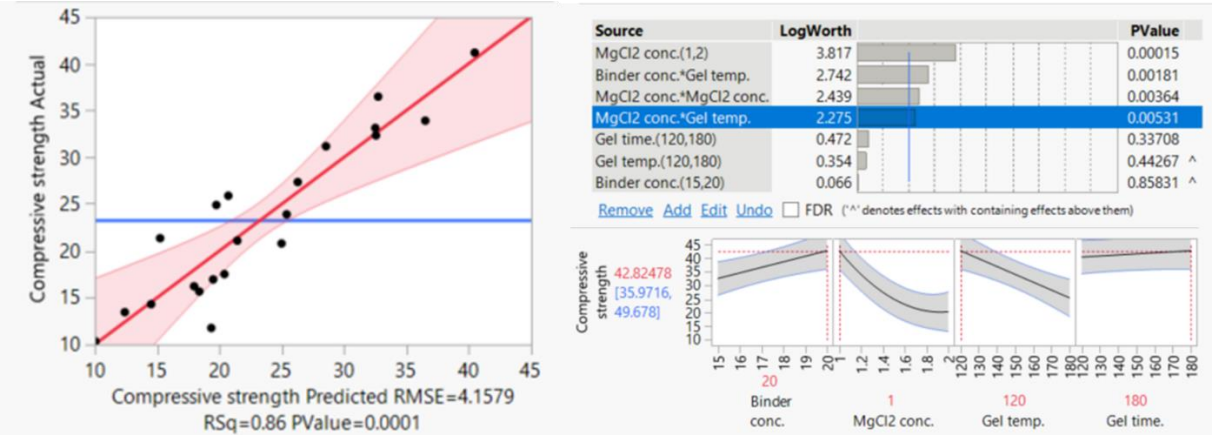
Table showing run combinations, run order, and resulting UCS

Run	**Effective starch conc. (wt%)**	**$MgCl_2$ conc. (M)**	**Gel. temp (°C)**	**Gel. time (min)**	**UCS (MPa)**
1	20	1.5	180	120	14.30
2	15	2	180	120	31.18
3	15	1	180	120	33.14
4	15	1.5	180	180	27.36
5	15	1.5	120	120	13.44
6	17.5	1.5	150	120	15.65
7	20	1	120	120	41.19
8	20	2	180	180	21.09
9	17.5	1.7	120	150	21.35
10	17.5	2	150	150	16.94
11	17.5	1	120	150	33.92

12	15	2	120	180	10.34
13	20	1.5	120	180	20.79
14	20	1.5	150	150	24.90
15	17.5	1.5	150	180	25.89
16	15	1.5	150	150	11.76
17	20	1	180	180	23.90
18	17.5	1	150	150	36.49
19	15	1	120	180	32.34
20	20	2	120	120	16.21
21	17.5	1.5	180	150	17.51

An RSM model was again constructed using the default settings as was done for the previous experiment. Effects with a low contribution towards the final model were again removed in order of significance (starting from least significant), until the LogWorth value for the least significant second order interaction (*i.e.*, $MgCl_2$.*gel. temp, highlighted below) was above a value of 1.

Although lower effective starch concentrations, higher $MgCl_2$ concentrations and higher gelatinisation temperatures had been predicted to improve UCS from previous experiments, the results from this experiment found the opposite to be true. This suggested that the optimal conditions had already been identified, and pushing the factor to more extreme levels did not improve the UCS. An increased gelation time from 120 min to 180 min marginally improved UCS, but it appeared to have largely plateaued after 120 minutes.



Details of resulting response surface model (RSM) and its predictions

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