The Mechanical Properties of Select Formulations of Pykrete Augmented with an Emulsifier in Uniaxial Compression

ENGR 059
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Abstract:

The mechanical properties of several different formulations of a cellulose fiber and frozen water composite called Pykrete were investigated through Uniaxial compression testing of standard three inch diameter compression testing cylinders in accordance with ASTM C-39-08 and ASTM C-873-08 [1,2]. Paper dust and saw dust were used as sources of cellulose fibers and the polysaccharide emulsifier xanthan gum was employed to keep these additives in suspension during freezing. Results indicate that several formulations of Pykrete are as strong as concrete in compression and even have higher strength to weight ratios.

Introduction:

Since the rapid development of Pykrete during World War II, there has been relatively little study of the material. Though its original application was as a naval construction material is no longer in need, it still has potential as a structural material that could be used in arctic environments for the construction of research outposts. The focus of this study was to determine the baseline mechanical properties of several formulations of Pykrete through compression testing of cylindrical specimens in accordance with ASTM C-39-08 and ASTM C-873-08 [1,2] and to compare these properties to those of concrete.

Background and Theory:

A Brief History of Pykrete and its Applications

Pykrete was developed during World War II as a cheap, strong, and ballistic-resistant material for the construction of a large aircraft carrier. At the time, the Allies' were hampered significantly by the short range of their aircraft [3]. Having a virtually indestructible mobile airbase would have been a serious tactical advantage. The idea of using ice as the main structural material for the enormous craft was originated by Geoffrey Pyke, who suggested modified icebergs as the base structure of a mobile air base [3]. In 1943, a scientist working at Brooklyn Polytechnic discovered that adding wood pulp to water and freezing it yielded a dramatically strengthened ice alloy that he called Pykrete, in honor of Geoffrey Pyke [3]. By the time the Allies determined an effective method of producing enough Pykrete to build a ship, aircraft technology had advanced to the point that a Pykrete aircraft carrier was no longer needed [3].

The Structure and Properties of Ice

Ice has a hexagonal planar structure that leads to an overall layered structure [4]. In slow loading, these layers slide over each other, but during faster loading, brittle fracture occurs [4]. These deformation behaviors are dependent to some degree on temperature: at lower temperatures, brittle fracture occurs regardless of loading speed [4]. The addition of fibrous additives, such as wood pulp, can combat the slipping of these crystalline planes by bridging the slip plane and can lead to an increase in strength of the ice alloy [4].

Additive Materials

Paper dust (Figure 1) was acquired from a local toilet paper factory and saw dust (Figure 2) was gathered from the table saw in the Engineering Department's shop.

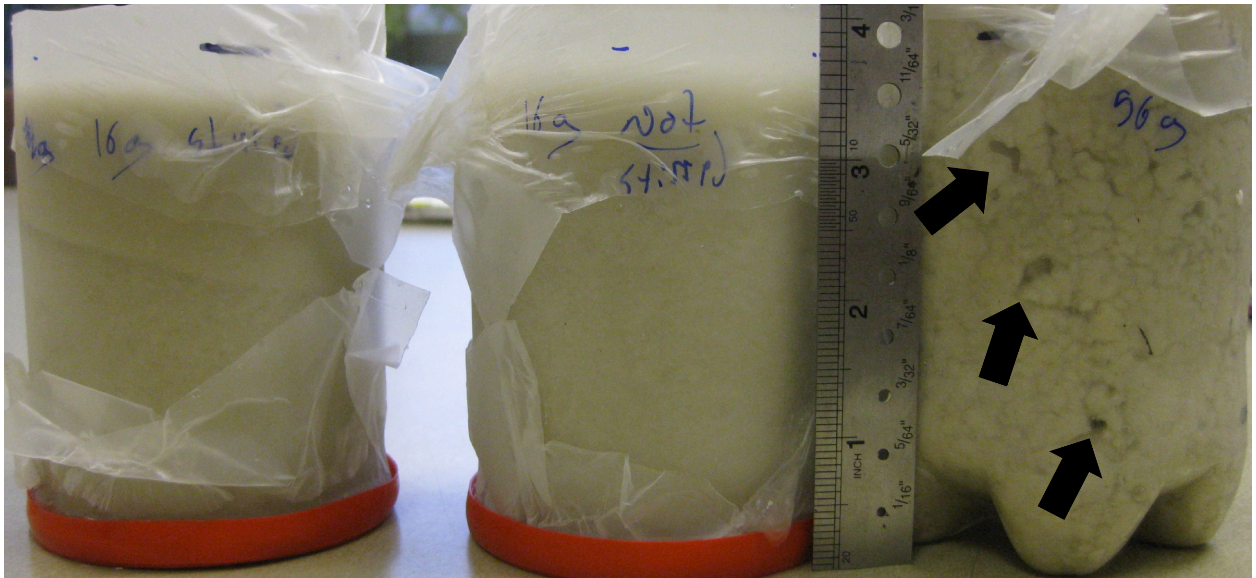

Figure 1 (left) and Figure 2 (right). Paper dust and saw dust.

Preliminary Experiments:

In order to avoid irregularities in mixing procedure or in the final Pykrete compression samples, two preliminary experiments were undertaken to evaluate likely problems that could be encountered when formulating the compression specimens.

The Effect of Mixing Technique on Additive Clumping and Dispersion of Paper Dust

The objective of the first experiment was to evaluate the impact of mixing technique on the distribution of paper dust and saw dust in water. Three 400 mL samples were prepared: one 4% by weight paper dust that had been thoroughly mixed with an Aluminum rotary mixer after the addition of water to the weighed paper dust, one 4% by weight paper dust that had been only been stirred enough to incorporate the paper dust into the water (labeled "not stirred"), and one 16% by weight paper dust that had not been stirred (stirring proved nearly impossible due to the shear thickness of the mixture and its resistance to yielding to a stirring implement). The samples were allowed to sit for twenty-four hours and were then were visually inspected for homogeneity, air pockets, and other signs of an uneven suspension (Figure 3).



4% BW PD, rotary mixer	4% BW PD, minimal mixing	16% BW PD, unmixed

Figure 3. The results of a preliminary mixing technique experiment. As indicated by the arrows, water-voids and air pockets were widespread throughout the 16% by weight paper dust sample. Though it is a subtler effect, there is additionally some settling of fibers in the 4% by weight samples.

The results indicated that a standardized mixing procedure was indeed necessary to thoroughly incorporate the paper dust and to eliminate air pockets and water-filled voids. Standardized and effective mixing was accomplished with the fabrication and use of an Aluminum rotary mixer, designer to be used at high speeds with an electric drill (Figure 4). The rotary mixer was sized to fit inside standard three-inch diameter concrete cylinder molds.

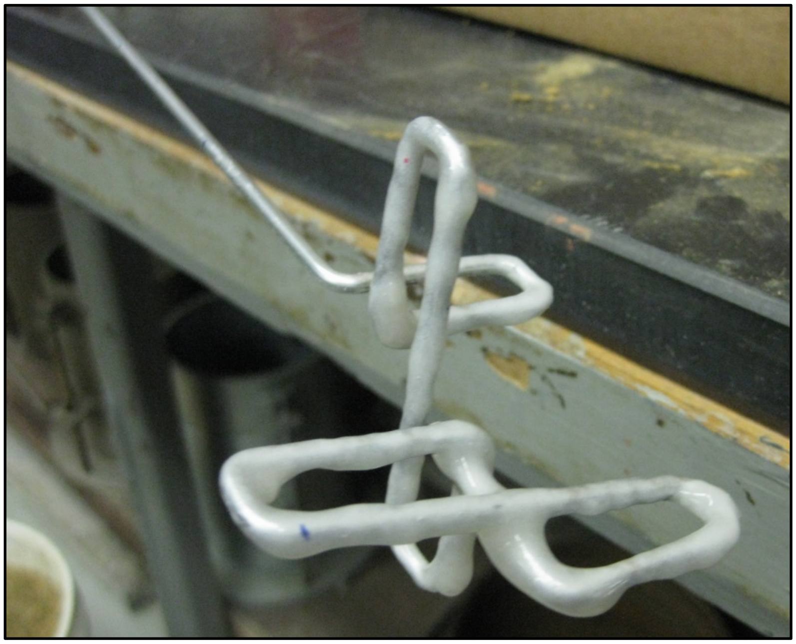


Figure 4. Aluminum rotary mixer fabricated to standardize and ensure good mixing of Pykrete samples.

The Effect of an Emulsifier upon the Homogeneity and Susceptibility to Additive Settling

As settling was determined early on to be a problem, emulsifiers, surfactants, and thickening agents were explored as possible methods to combat this problem. Early on, agarose gel was investigated as a gelling compound that would guarantee an even suspension of fibers by forming a relatively rigid gel containing the fibrous additive. Unfortunately, agarose requires high temperatures in order to be dissolved in water, making it an impractical ingredient for large samples of Pykrete or potential use in the field. Further research led to the discovery of xanthan gum, a polysaccharide used in the food industry as a thickener and emulsifier. Xanthan gum not only did not require heating to be dissolved in water, but it had unique viscosity properties: at low rates of shear, xanthan gum solutions are quite viscous, but at higher rates of shear, their viscosity drops significantly [5]. This property made xanthan gum an ideal compound for keeping the fibrous additives in suspension.

Experiments were carried out to determine the efficacy and required concentration of xanthan gum. Three samples were fabricated: one 4% by weight paper dust sample, one 4% by weight saw dust sample, and one 16% by weight saw dust sample. Each was mixed with the rotary mixer and was then allowed to sit for several minutes until settling had been observed. Then the first xanthan gum concentration was added: 0.5% of the sample's weight in xanthan gum, which was then mixed into solution with the rotary mixer. As can be seen below (Figures 5&6), the results were striking.

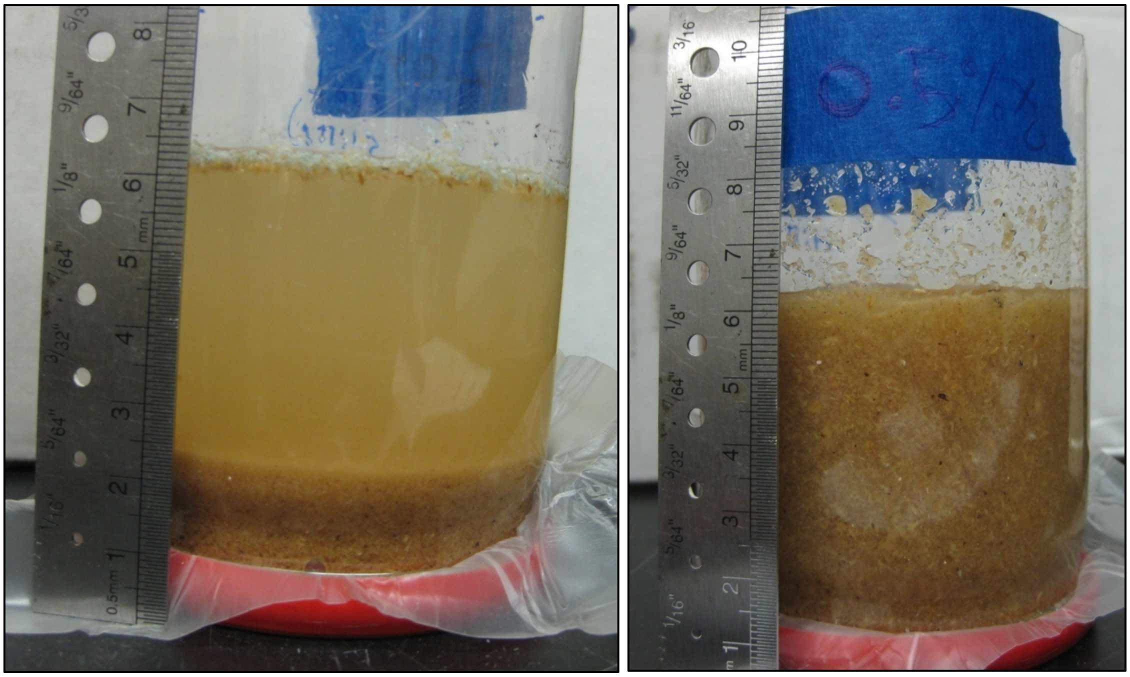


Figure 5. The effect of 0.5% by weight xanthan gum on a 4% by weight saw dust solution. Left: without the addition of xanthan gum. Right: With the addition of xanthan gum.

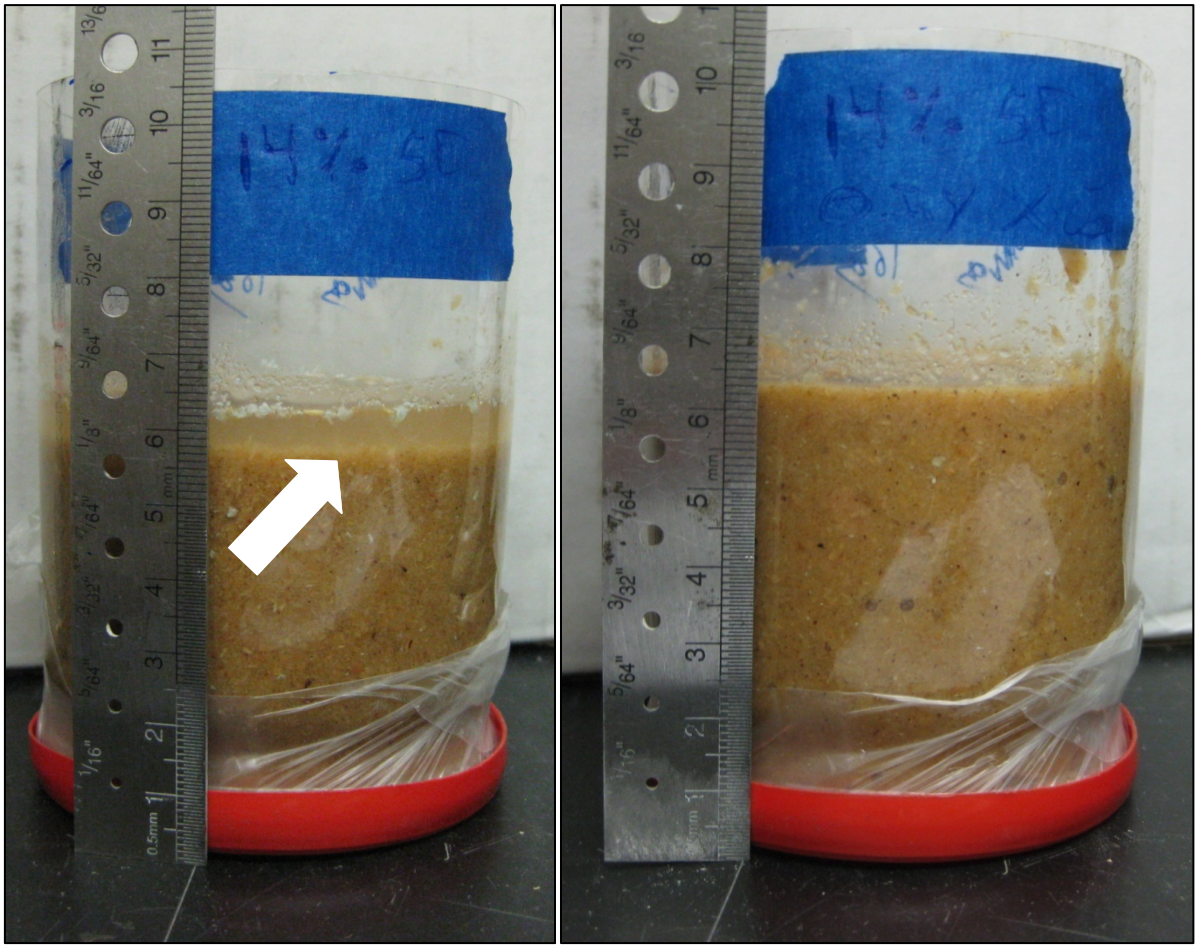


Figure 6. The effects of 0.5% by weight xanthan gum on a 16% by weight saw dust solution. Left: without the addition of xanthan gum. Right: with the addition of xanthan gum.

Based on these results, it was evident that a 0.5% by weight concentration of xanthan gum was entirely sufficient to keep the additive materials suspended until the solution froze. Efforts to determine the impact of higher concentrations of xanthan gum were abandoned due to these results.

Experimental Methods:

Four different formulations and two controls were fabricated, as shown below.

	Paper Dust	Saw Dust
4% By Weight	3	3
14% By Weight	3	3

Table 1. Number of formulations fabricated.

In order to contextualize the results of the Pykrete compression tests and to determine the baseline compressive strength of pure ice, three water controls were made. Additionally, three 0.5% xanthan gum solution controls were made to study the effects of xanthan gum alone on the mechanical properties of ice.

All components of a given formulation were weighed into the sample's mold on a tared gram balance. For low weight concentration samples, the appropriate amount of water was added to the mold and then the contents were mixed at high speed with the rotary mixer. More viscous samples were individually weighed, dry mixed, and then combined in a large vessel to which water for all three samples was added. The mixture was then thoroughly blended with the rotary mixer and divided equally among three molds. This procedure was employed for the xanthan gum controls because of clumping in the xanthan gum powder after the addition of water. Once a sample was completely mixed, it was capped with aluminum foil and the perimeter was taped to seal the sample. Samples were carefully transported to the Sharples dining hall ice cream freezer, which is maintained at a temperature between -10 and 0 degrees Fahrenheit. After freezing, each sample's mold was scored with a band saw to facilitate easy removal of the molds with a de-molding tool and the sample was kept on a bed of dry ice.

Experimental Apparatus:

The Engineering Department's hydraulic universal testing machine (Figure 7) was used for compression testing. Steel end caps with rubber seats were used to distribute the load across the ends of each sample. The end caps were brought to temperatures well below freezing by keeping them on dry ice in between compression tests. Sample deflection measurements were taken by measuring the distance travelled by an Allen wrench that was secured to the loading head by a hose clamp.

Figure 7. Hydraulic universal testing machine. A digital read out was used to display loading of the samples and a dial gauge was used record axial deflection of the sample.

Results:

All formulations of Pykrete demonstrated increased compressive strength over the pure water controls, which could not bear the small stresses involved in the de-molding process and shattered before any controlled testing could be performed. Though strain data was collected for many samples, the stress strain plots demonstrated that though certain regions are roughly linear, it is very difficult to accurately determine the modulus of elasticity of the samples (Figures 8-10). Additionally, the xanthan gum control samples demonstrated increased compressive strength over the pure water control samples.

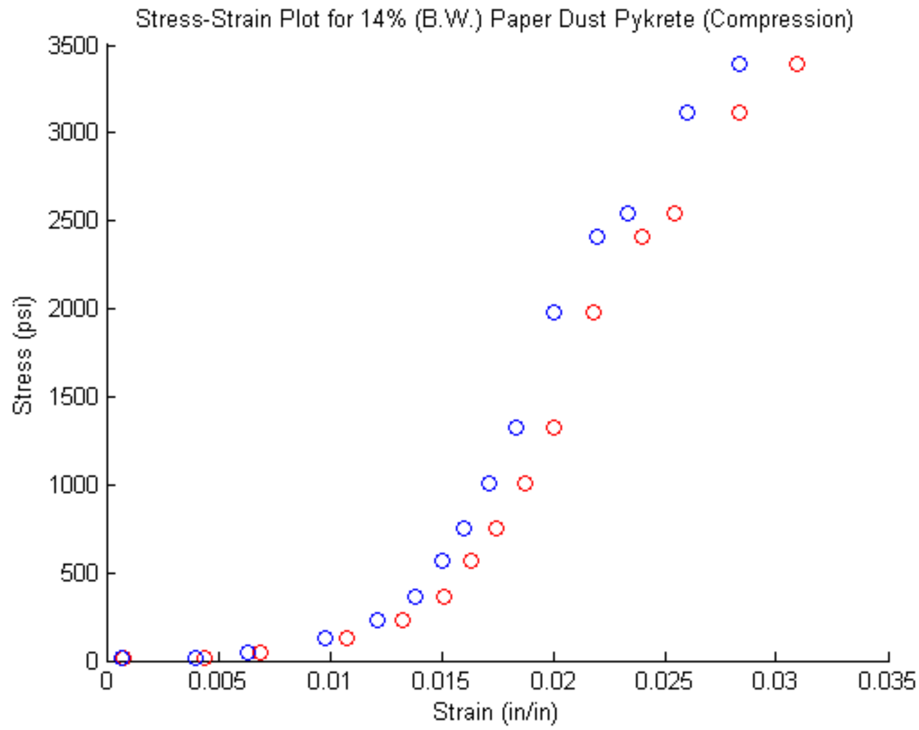


Figure 8. Stress and strain of a 14% by weight paper dust sample in compression.

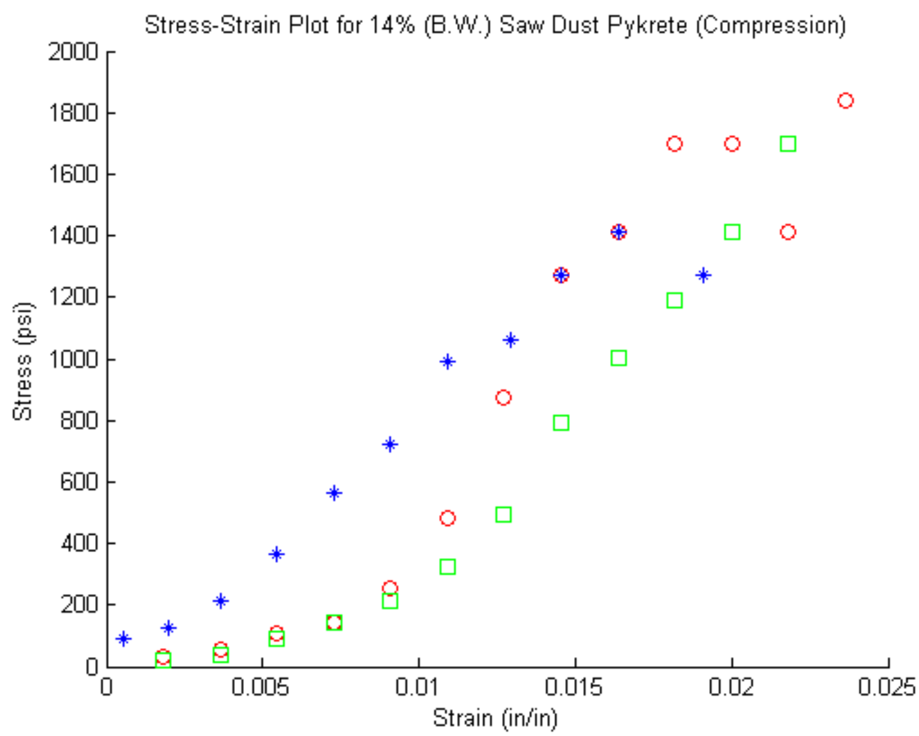


Figure 9. Stress and Strain for three samples of 14% by weight saw dust. Alternate strain values were not included in this plot for sake of clarity.

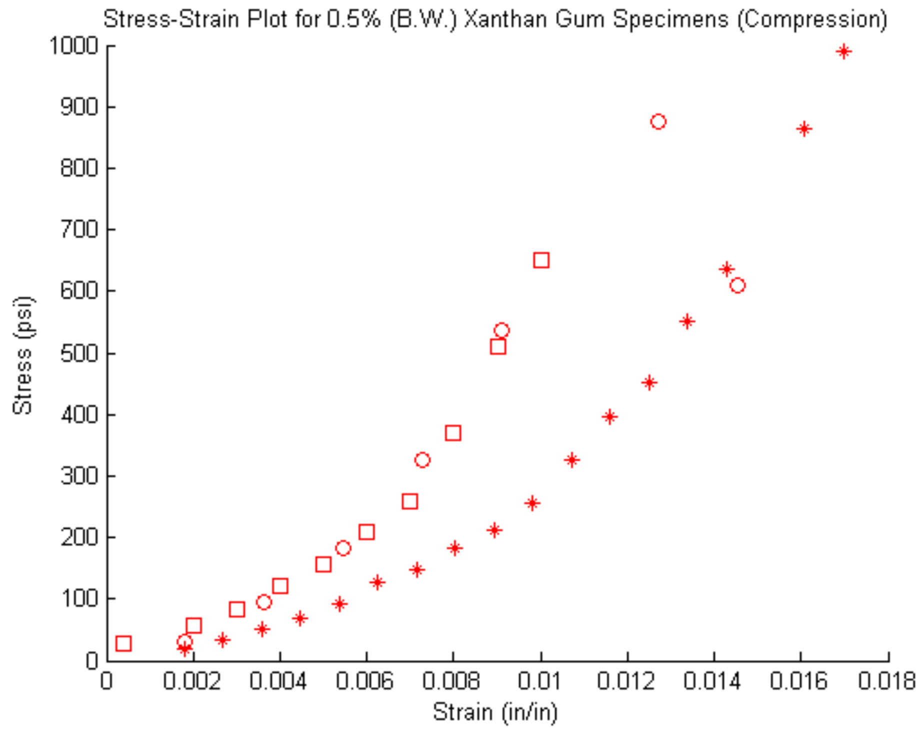


Figure 10. Stress and strain for 0.5% by weight xanthan gum controls.

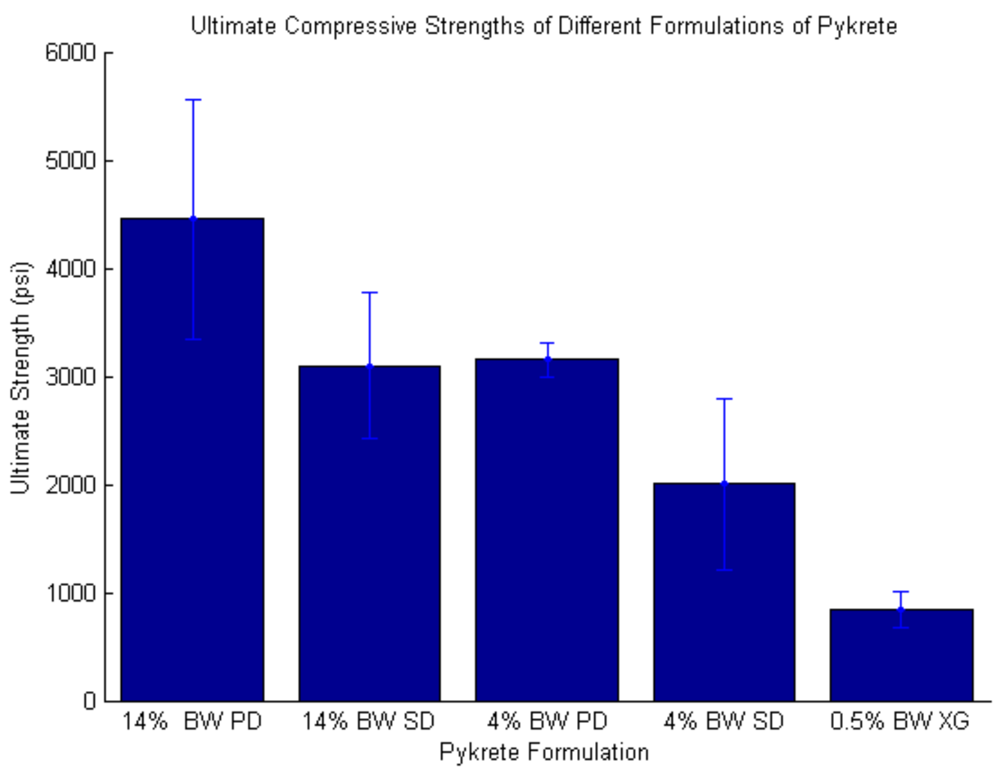


Figure 11. Ultimate strengths of all Pykrete formulations studied.

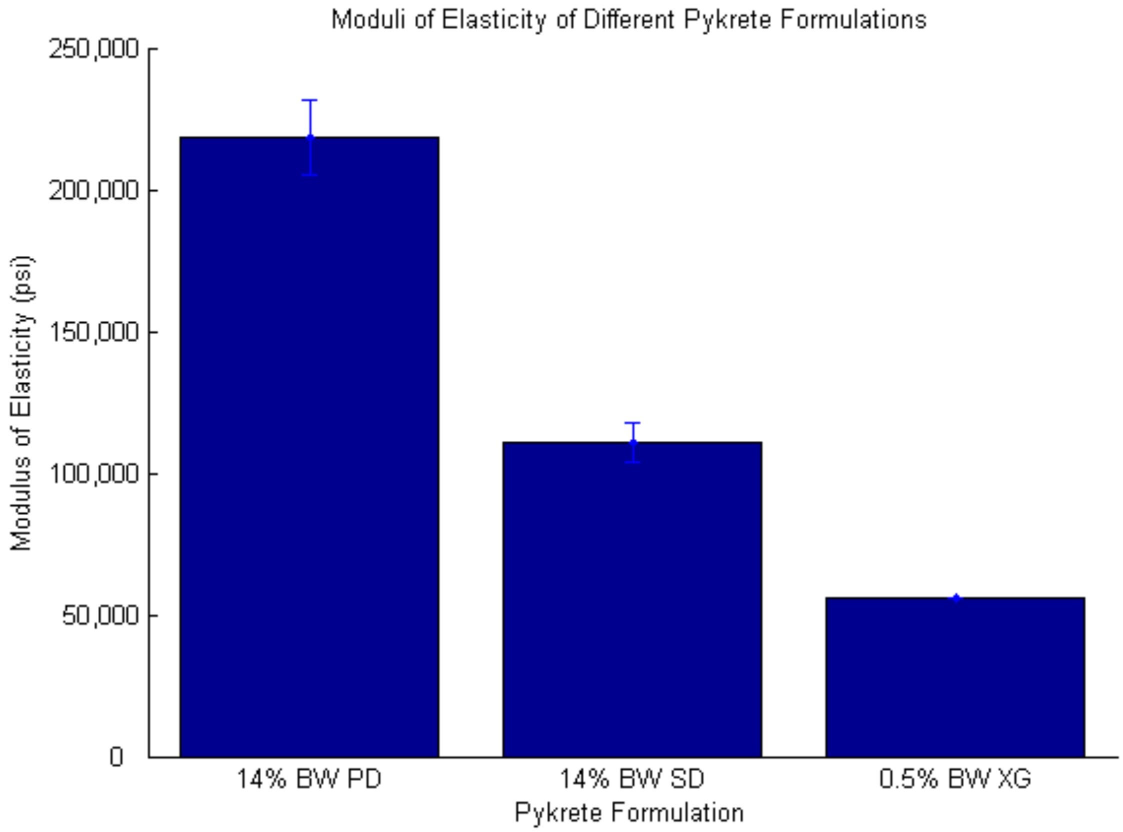


Figure 12. Modulus of elasticity of different formulations of Pykrete.

Discussion:

Testing goals and methods varied over the course of the project, as is detailed in the below table (Table 2).

	Initial Length Measured	Displacement Data Recorded	Tested to Ultimate Strength	Tested for Impact Resistance
4% BW Paper Dust	N	N	Y	N
14% BW Paper Dust	N	For one sample	Y	N
4% BW Saw Dust	N	N	Y	N
14% BW Saw Dust	N	Y	Y	N
0.5% BW Xanth. Gum	Y	Y	Y	N
Water Control	-	-	-	-

Table 2. Testing procedures for different samples.

Because the initial length of most samples was not recorded, all strain data for these samples is approximate. All samples were between 5.5 inches and 6.0 inches tall, so it is possible to bound the possible strain values with these lengths, as is demonstrated in Figure 8. Given that the strain data is a range rather than a distinct value, calculated the modulus of elasticity for the different samples incorporate this uncertainty. Additionally, when compared to the modulus of elasticity of concrete, calculated values are significantly smaller, indicating either the incomparability of the two materials or the unreliability of the calculated the modulus of elasticity (Table 3 and Figure 12).

	Concrete	PD 14%	SD 14%	XG.0.5%
Modulus of Elasticity (GPa)	30	1.5	0.76	0.39

Table 3. Comparison of the modulus of elasticity of different Pykrete samples to that of concrete [7].

Though strain values for most samples are questionable, stress values are more accurate and demonstrate the strength of the material well. The behavior of the material under compression was somewhat dependent on the rate of loading. At higher loads, the samples continuously deflected at a rate of two ten-thousandths per second. In the case of the 14% by weight paper dust samples, failure was difficult to define because samples never lost significant structural integrity and just deflected more and more rapidly as the load increased. Ultimate strength was based on the highest load that the sample would consistently bear at a rapid rate of loading. All other samples failed explosively and ultimate strength was based on the maximum load achieved before this dramatic failure. As can be seen in Table 4 and Figure 11, the ultimate strength of certain formulations of Pykrete is comparable to that of concrete.

	Concrete	PD 14%	PD 4%	SD 14%	SD 4%	XG.0.5%
Density (g/cc)	2.4	~1	~1	~1	~1	~1
Compressive Strength (psi)	2500-5000	4453	3097	3155	2002	840
Compressive Strength (MPa)	16-34	30.7	21.4	16.6	13.8	5.8
Strength to Weight Ratio (kN/kg)	6.7-14.2	~30.7	~21.4	~16.6	~13.8	~5.8

Table 4. Comparison of compressive strengths of different Pykrete formulations to that of concrete and comparison of strength to weight ratios [7].

Finally, impact testing was planned for all four formulations of Pykrete and the two control groups, but time constraints prevented this dimension of testing.

Conclusions:

Though there were inconsistencies in testing methods and strain data collected was not as accurate as would be desired, this investigation of the mechanical properties of Pykrete has yielded several important conclusions:

- Pykrete formulated with paper dust is significantly stronger than that formulated with saw dust (Table 4, Figure 11).
- Pykrete that is 14% by weight paper dust is just as strong as some concretes and has a strength to weight ratio of up to four times that of concrete (Table 4).
- Some formulations of Pykrete do not fail explosively like many concretes, giving them potential in applications where safety and warning of failure are important.
- The addition of xanthan gum to solutions of low weight percent paper dust and saw dust allows for much higher ultimate strengths than Pykrete formulated with plain water (Table 4, Figure 11).
- The components of Pykrete require significantly less processing than those of concrete.

There is much more room for exploration of this fascinating material.

Acknowledgements:

I like to give thanks to Professor Siddiqui and Grant Smith for their aid in setting up the testing machines and for their suggestions regarding my experimental methods, to Kanti Somani for procuring large quantities of paper dust, to Donna Halley for her aid in acquiring dry ice, and to Stacey Daugherty for her early consultations on gelling agents.

References:

[1] ASTM C-39-08: Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens.

[2] ASTM C-873-08: Standard Test Method for Compressive Strength of Concrete Cylinders Cast in Place in Cylindrical Molds.

[3] Perutz, M. F. 1946, "A Description of the Iceberg Aircraft Carrier," *Journal of Glaciology*, Vol. 1, No. 2, p. 51.

[4] W. D. KingerySource, 1961, "Ice Alloys," Science, New Series, Vol. 134, No. 3473.

[5] JA Casas, AF Mohedano, F Garcia-Ochoa, 2000, "Viscosity of guar gum and xanthan/guar gum mixture solutions,"J. Sci. Food Agric. Vol. 80, pp. 1722-1727.

[6] Engineering ToolBox, 2010, "Elastic Properties and Young's Modulus for Some Materials," online article, http://www.engineeringtoolbox.com/young-modulus-d_417.html.

[7] Brady G.S., Clauser H.R., 1991, "Materials Handbook: An Encyclopedia for Managers, Technical Professionals, Purchasing and Production Managers, Technicians, Supervisors, and Foremen," McGraw-Hill, Inc., pp. 255-256.

Appendices

Raw Data Spreadsheets:

Xanthan Gum Control Samples

xg-0.5-1			Lo = 5.5
displacement	load	stress	strain
10	220	31.12363	0.001818
20	680	96.20032	0.003636
30	1300	183.9124	0.005455
40	2300	325.3834	0.007273
50	3800	537.59	0.009091
80	4300	608.3256	0.014545
70	6200	877.1206	0.012727

xg-0.5-2			Lo = 5.6
displ	load	stress	strain
10	140	19.80595	0.001786
15	240	33.95305	0.002679
20	360	50.92958	0.003571
25	490	69.32082	0.004464
30	650	91.95619	0.005357
35	90	12.7324	0.00625
40	1050	148.5446	0.007143
45	1300	183.9124	0.008036
50	1500	212.2066	0.008929
55	1800	254.6479	0.009821
60	2300	325.3834	0.010714
65	2800	396.119	0.011607
70	3200	452.7074	0.0125
75	3900	551.7371	0.013393
80	4500	636.6198	0.014286
90	6100	862.9735	0.016071
95	7000	990.2974	0.016964

xg-0.5-3			Lo = 5.0
displ	load	stress	strain
2	200	28.29421	0.0004
10	400	56.58842	0.002
15	580	82.05322	0.003
20	850	120.2504	0.004
25	1110	157.0329	0.005
30	1480	209.3772	0.006
35	1830	258.892	0.007

40	2620	370.6542	0.008
45	3600	509.2958	0.009
50	4600	650.7669	0.01

14% By Weight Paper Dust Samples

pd-14-1

vid				Lo=6
load	deflection	stress	strain5.5	strain6
140	4	19.80595	0.000727	0.000667
110	24	15.56182	0.004364	0.004
300	38	42.44132	0.006909	0.006333
900	59	127.324	0.010727	0.009833
1600	73	226.3537	0.013273	0.012167
2600	83	367.8248	0.015091	0.013833
4000	90	565.8842	0.016364	0.015
5300	96	749.7966	0.017455	0.016
7100	103	1004.445	0.018727	0.017167
9400	110	1329.828	0.02	0.018333
14000	120	1980.595	0.021818	0.02
17000	132	2405.008	0.024	0.022
18000	140	2546.479	0.025455	0.023333
22000	156	3112.363	0.028364	0.026
24000	170	3395.305	0.030909	0.028333
peak load:	37000	5234.429		

4% By Weight Paper Dust Samples

	Ultimate Load (lbs)	Stress (psi)	Notes
PD-4-1	24280	3434.917	explosive
PD-4-2	16410	2321.54	non-explosive--slumped and slower loading
PD-4-3	24990	3535.362	explosive

14% By Weight Saw Dust Samples

sd-14-1 some small exterior voids

deflection	load	stress	strain5.5	strain6
10	220	31.12363	0.001818	0.001667
20	400	56.58842	0.003636	0.003333
30	750	106.1033	0.005455	0.005
40	1030	145.7152	0.007273	0.006667
50	1800	254.6479	0.009091	0.008333
60	3400	481.0016	0.010909	0.01
70	6200	877.1206	0.012727	0.011667
80	9000	1273.24	0.014545	0.013333

90	10000		1414.711	0.016364	0.015
100	12000		1697.653	0.018182	0.016667
110	12000	displ decr steadily	1697.653	0.02	0.018333
120	10000		1414.711	0.021818	0.02
130	13000		1839.124	0.023636	0.021667
ult load	22830		3229.784		

sd-14-2

displ	load	stress	strain5.5	strain6
3	660	93.3709	0.000545	0.0005
11	900	127.324	0.002	0.001833
20	1500	212.2066	0.003636	0.003333
30	2600	367.8248	0.005455	0.005
40	4000	565.8842	0.007273	0.006667
50	5100	721.5024	0.009091	0.008333
60	7000	990.2974	0.010909	0.01
71	7500	1061.033	0.012909	0.011833
80	9000	1273.24	0.014545	0.013333
90	10000	1414.711	0.016364	0.015
105	9000	1273.24	0.019091	0.0175
failure:	21000	2970.892		

sd-14-3

displ	load	stress	strain5.5	strain6
10	140	19.80595	0.001818	0.001667
20	250	35.36777	0.003636	0.003333
30	650	91.95619	0.005455	0.005
40	1000	141.4711	0.007273	0.006667
50	1500	212.2066	0.009091	0.008333
60	2300	325.3834	0.010909	0.01
70	3500	495.1487	0.012727	0.011667
80	5600	792.2379	0.014545	0.013333
90	7100	1004.445	0.016364	0.015
100	8400	1188.357	0.018182	0.016667
110	10000	1414.711	0.02	0.018333
120	12000	1697.653	0.021818	0.02
failure	23070	3263.737		

4% By Weight Saw Dust Samples

	ult load	stress	
sd-4-1	8240	1165.722	splintery failure--slower loading rate
sd-4-2	14830	2098.016	explosive failure
sd-4-3	19390	2743.124	very explosive failure

MATLAB Code Written to Process Data and Generate Plots:

```
% MATLAB code for analysis and plotting of data for ENGR 59
% project.
%
% Andreas Bastian
% December 2010

%Saw dust 14% by weight stress strain data:
clear all;
load('data.mat');
hold on;
plot(pd141(:,2),pd141(:,1),'ro');
plot(pd141(:,3),pd141(:,1),'bo');
xlabel('Strain (in/in)');
ylabel('Stress (psi)');
title('Stress-Strain Plot for 14% (B.W.) Paper Dust Pykrete (Compression)')
hold off;
pdFit = polyfit(-pd141(5:end-1,2),-pd141(5:end-1,1),1);

%% Saw dust 14% by weight stress strain data:
load('data.mat');
%Saw dust 14% by weight stress strain data:
hold on;
plot(sd141(:,2),sd141(:,1),'ro');
% plot(-sd141(:,3),-sd141(:,1),'ro');
plot(sd142(:,2),sd142(:,1),'b*');
% plot(-sd142(:,3),-sd142(:,1),'b*');
plot(sd143(:,2),sd143(:,1),'gs');
% plot(-sd143(:,3),-sd143(:,1),'gs');
xlabel('Strain (in/in)');
ylabel('Stress (psi)');
title('Stress-Strain Plot for 14% (B.W.) Saw Dust Pykrete (Compression)');
hold off;
sdFit1 = polyfit(-sd141(5:end-1,3),-sd141(5:end-1,1),1);
sdFit2 = polyfit(-sd142(5:end-1,3),-sd142(5:end-1,1),1);
sdFit3 = polyfit(-sd143(5:end-1,3),-sd143(5:end-1,1),1);
modEavg = [sdFit1(1),sdFit2(1),sdFit3(1)]; %avg modulus of Elas
modEavg = mean(modEavg);

%% Xanthan Gum 0.5% by weight controls
load('data.mat');
hold on;
plot(xg051(:,2),xg051(:,1),'ro');
plot(xg052(:,2),xg052(:,1),'r*');
plot(xg053(:,2),xg053(:,1),'rs');
xlabel('Strain (in/in)');
```

```
ylabel('Stress (psi)');
title('Stress-Strain Plot for 0.5% (B.W.) Xanthan Gum Specimens
(Compression)')
hold off;
xgFit1 = polyfit(xg051(5:end-1,2),xg051(5:end-1,1),1);
xgFit2 = polyfit(xg052(5:end-1,2),xg052(5:end-1,1),1);
xgFit3 = polyfit(xg053(5:end-1,2),xg053(5:end-1,1),1);
modEavg = [xgFit1(1),xgFit2(1),xgFit3(1)]; %avg modulus of Elas
modEavg = mean(modEavg);
%% Bar graph comparing moduli of elasticity amongst materials
pd14 = [2.2785e5, 2.0886e5];
sd14 = [1.0608e5, 1.1592e5];
xg05 = 5.5932e4;
pd14m = mean(pd14);
pd14stdev = std(pd14);
sd14m = mean(sd14);
sd14stdev = std(sd14);
x = [pd14m, sd14m, xg05];
hold on;
bar(x);
errorbar(x,[pd14stdev,sd14stdev, 0],'.');
xlabel('Pykrete Formulation');
ylabel('Modulus of Elasticity (psi)');
title('Moduli of Elasticity of Different Pykrete Formulations');
%% Bar graph comparing average ultimate strength
sd4 = [1165.7,2098, 2743.1];
pd14 = [5234.4,3671.2];
pd4 = [3434.9,2321.5,3535.4];
sd14 = [3230.0,2970.9,3263.7];
xg05 = [877.1,990.3,651.0];
hold on;
x = [mean(pd14),mean(pd4),mean(sd14),mean(sd4),mean(xg05)];
bar(x);
errorbar(x,[std(pd14),std(pd4),std(sd14),std(sd4),std(xg05)],'.');
xlabel('Pykrete Formulation');
ylabel('Ultimate Strength (psi)');
title('Ultimate Strengths of Different Formulations of Pykrete');
```