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**ISO 13503-2 SUITE  
BASELINE FRACTURE CONDUCTIVITY &  
PERMEABILITY  
CERAMIC PROPPANT**

**ISP (30/50) Yuanqu Corundum Proppants Corporation**

**SUPPLIED BY CMC COMETALS CHINA**

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## **SUMMARY**

A sample of **ISP (30/50)** proppant was provided for testing by CMC COMETALS China, manufactured by Yuanqu Corundum Proppants Corporation.

Testing was carried out in accordance with the ISO 13503-2 suite of tests covering crush resistance, acid solubility, bulk density, sphericity and roundness, turbidity and sieve analysis. Specific gravity testing was also performed.

The baseline conductivity and permeability tests of the sample were performed with a loading of 2lb/ft<sup>2</sup>, at closure stresses of 2000, 4000, 6000, 8000, 10000 and 12000psi, at 250°F, each maintained for 50 hours between Ohio sandstone.

A summary of results obtained from the ISO 13503-2 suite of tests is tabulated overleaf. Long term baseline conductivity results are presented in the main report.

Photomicrographs are given in Appendix A.

Parameters		ISP (30/50)
Crush Test (psi)	10000	1.6 ± 0.4%
	12500	3.1 ± 0.6%
Sieve Analysis (90% 30/50)		94.2 ± 1.3
Sieve Analysis (0.1% -70)		0.0 ± 0.0
Sieve Analysis (1% +20)		0.0 ± 0.0
Mean Size (mm)		0.514 ± 0.006
Bulk Density (g/cm <sup>3</sup> )		1.79 ± 0.00
Specific Gravity		3.25 ± 0.03
Acid Solubility (%)		6.4 ± 0.4
Sphericity		0.9 ± 0.0
Roundness		0.9 ± 0.1
Turbidity (FTU)		30 ± 4

*Table i: Summary of API RP 60 & ISO 13503-2 Test Suite on Proppant Samples*

## 1. EXPERIMENTAL PROCEDURES

### 1.1 Specific Gravity

A dry density bottle, with stopper, is weighed ( $W_1$ ). Approximately 30g of proppant is added and the bottle re-weighed ( $W_2$ ). Mineral oil is added to cover the proppant and fill the bottle 3/4 full. The bottle is vacuumed to an absolute pressure of less than 30 mbar for approximately thirty minutes.

More oil is added to fill the bottle. The stopper is then placed in the neck. Excess oil is wiped off, being careful not to suck oil through the capillary in the stopper. The full bottle is weighed ( $W_3$ ). The proppant and oil are disposed of and the bottle refilled with mineral oil only. The stopper is placed in the neck and the bottle dried and weighed again ( $W_4$ ). This procedure is repeated.

The Specific Gravity is then given by,

$$(W_2 - W_1) / ((W_4 - W_3) + (W_2 - W_1))$$

## 1.2 ISO 13503-2 Methods

The following tests were performed in exact accordance with the ISO 13503-2 procedures. The relevant sections are as follows:

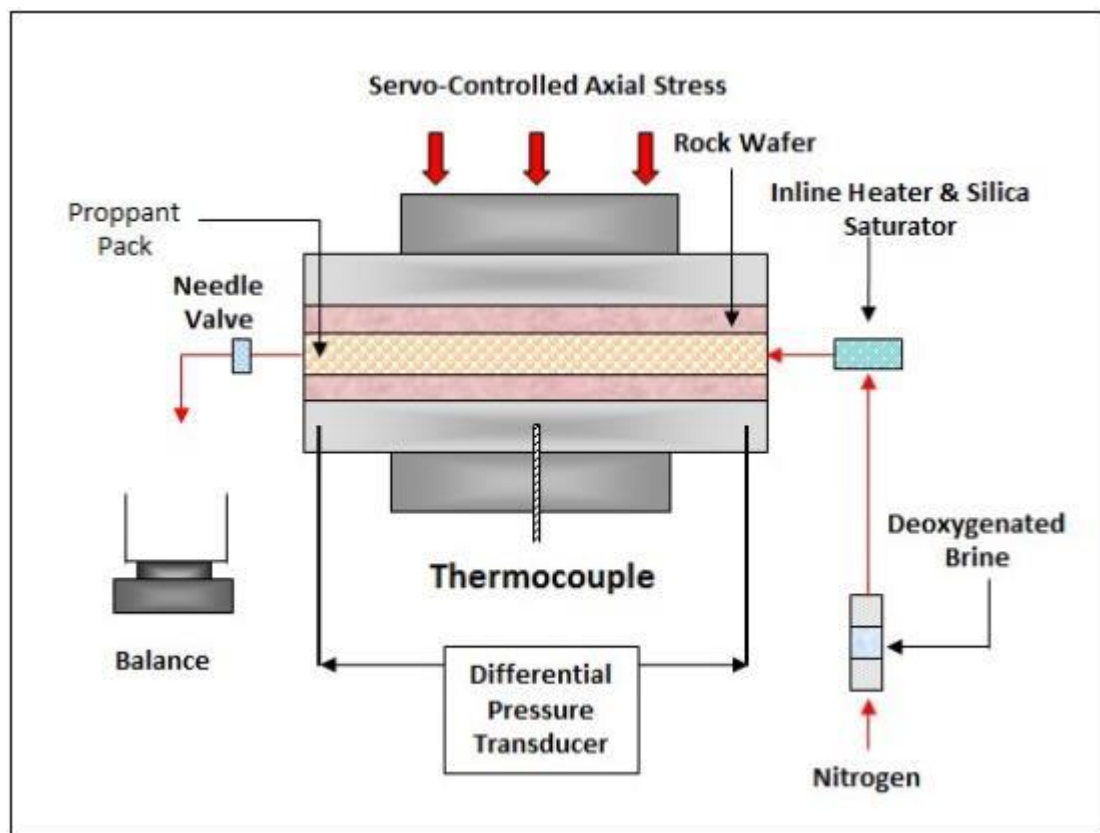
Section	Test/Procedure
5.2	Sample Splitting
6	Sieve Analysis
7	Sphericity and Roundness
8	Acid Solubility (tests run using 60% HF as source)
9	Turbidity
10.3	Bulk Density
11	Crush Resistance (with pluviator)*

**Table 1: ISO Procedures**

\*Crush testing was carried out at three pressures, 10000 and 12500psi.

### 1.3 Baseline Fracture Conductivity and Permeability

The Fracture Conductivity Cells allow for samples of proppant of various loading to be subjected to closure stress and temperature over extended time. Fluids were flowed through the pack and, from differential pressure measurements, the flow capacity of the pack could be determined. A schematic of the experimental set-up is given below. The cell was essentially a modified 10 square inch API conductivity cell.



*Figure 1: Configuration of Conductivity Cell*

The test procedure is as follows:

- I. Outcrop rocks were selected. For these tests, Ohio Sandstone was used. Ohio Sandstone has a static elastic modulus of approximately 4 million psi and a permeability of ~1mD. Wafers of thickness 9.5mm were machined to 0.05mm precision and one rock was placed in the cell.
- II. The selected proppant was sample split and weighed out. Sample splitting ensured that a representative sample was achieved in terms of its particle size distribution.
- III. In cell 1, the proppant was placed into the cell and levelled. The top rock was then inserted.
- IV. Heated steel platens provided the correct temperature simulation for the test. A thermocouple inserted in the middle port of the cell wall recorded the temperature of the pack. A servo-controlled loading ram provided the closure stress.
- V. The cells were initially set at 80°F and 1000psi. The cells were then heated to 250°F and held for 15 hours at 1000psi before being ramped to 2000psi over 10 minutes. After 15 hours a set of measurements was made before the stress was ramped to 4000psi (total time: 115 hours).
- VI. Further measurements were made at 6000, 8000, 10000 and 12000psi at intervals of 50 hours, corresponding to a total time of 315 hours.



Conductivity measurements were made using the following procedure:

- I. A 70mbar full range differential pressure transducer was activated by closing the bypass valve and opening the low pressure line valve (the second valve was there to prevent fluid flow bypassing the cell itself while the d.p. bypass was open).
- II. When the differential pressure appeared to be stable, the output was fed to a data logger 5 digit resolution multi-meter which logged the output every second during measurement.
- III. Fluid was collected for 5 to 10 minutes. The flow rate was obtained from a precision stepper pump which accurately controlled and metered flow. The mean value of the differential pressure was retrieved from the multi-meter together with the peak high and low values. If the difference between the high and low values was greater than the 5% of the mean, the data was disregarded.
- IV. Temperature was recorded from the inline thermocouple at the start and end of the flow test period. If the temperature variation was greater than 0.5°K the test was disregarded. Viscosity of the fluid was obtained from using the measured temperature and viscosity tables. For brine at 100psi, no pressure correction was made. The density of brine at elevated temperature is obtained from these tables.
- V. At least three permeability determinations were made at each stage. The standard deviation of the determined permeabilities had to be less than 1% of the mean value for the test sequence to be considered acceptable.
- VI. At the end of the permeability testing, the widths of each of the four corners of the cell were determined using vernier callipers, to 0.01mm resolution.

## 2. RESULTS

### 2.1 Sieve Analysis

The result of the sieve analysis is tabulated below:

ASTM Mesh	ISP (30/50)			
	% Proppant (A) by weight	% Proppant (B) by weight	% Average by weight	Standard Deviation
+20	0.00	0.00	<b>0.0</b>	0.0
-20 +30	6.67	4.85	<b>5.8</b>	1.3
-30 +35	49.05	46.25	<b>47.7</b>	2.0
-35 +40	37.91	41.25	<b>39.6</b>	2.4
-40 +45	6.21	7.56	<b>6.9</b>	1.0
-45 +50	0.16	0.09	<b>0.1</b>	0.1
-50 +70	0.00	0.00	<b>0.0</b>	0.0
-70	0.00	0.00	<b>0.0</b>	0.0
<b>Total</b>	100.0	100.0	<b>100.0</b>	
<b>%30/50</b>	93.3	95.1	<b>94.2</b>	1.3
<b>Mean Size(mm)</b>	0.518	0.510	<b>0.514</b>	0.006

*Table 2: Sieve Analysis*

## 2.2 Bulk Density & Specific Gravity

The result of the bulk density and specific gravity testing is tabulated below

Sample	Bulk Density (g/cm <sup>3</sup> )	Standard Deviation (g/cm <sup>3</sup> )	Specific Gravity	Standard Deviation
ISP (30/50)	1.79	0.00	3.25	0.03

*Table 3: Bulk Density & Specific Gravity Results*

## 2.3 Acid Solubility

The results of the Acid Solubility are tabulated below:

Sample No.	Test No.	% Loss, by Mass	Average % Loss, by Mass	Standard Deviation % Loss, by Mass
ISP (30/50)	1	6.148	6.4	0.4
	2	6.702		

*Table 4: Acid Solubility Results*

## 2.4 Sphericity & Roundness

ISP (30/50)		
Sample Reference	Sphericity	Roundness
1	0.9	0.7
2	0.9	0.9
3	0.9	0.9
4	0.9	0.9
5	0.9	0.9
6	0.9	0.9
7	0.9	0.9
8	0.9	0.9
9	0.9	0.9
10	0.9	0.9
11	0.9	0.9
12	0.9	0.9
13	0.9	0.9
14	0.9	0.9
15	0.9	0.9
16	0.9	0.9
17	0.9	0.9
18	0.9	0.7
19	0.9	0.9
20	0.9	0.9
	<b>Mean = 0.9</b>	<b>Mean = 0.9</b>
	<b>Standard Deviation = 0.0</b>	<b>Standard Deviation = 0.1</b>

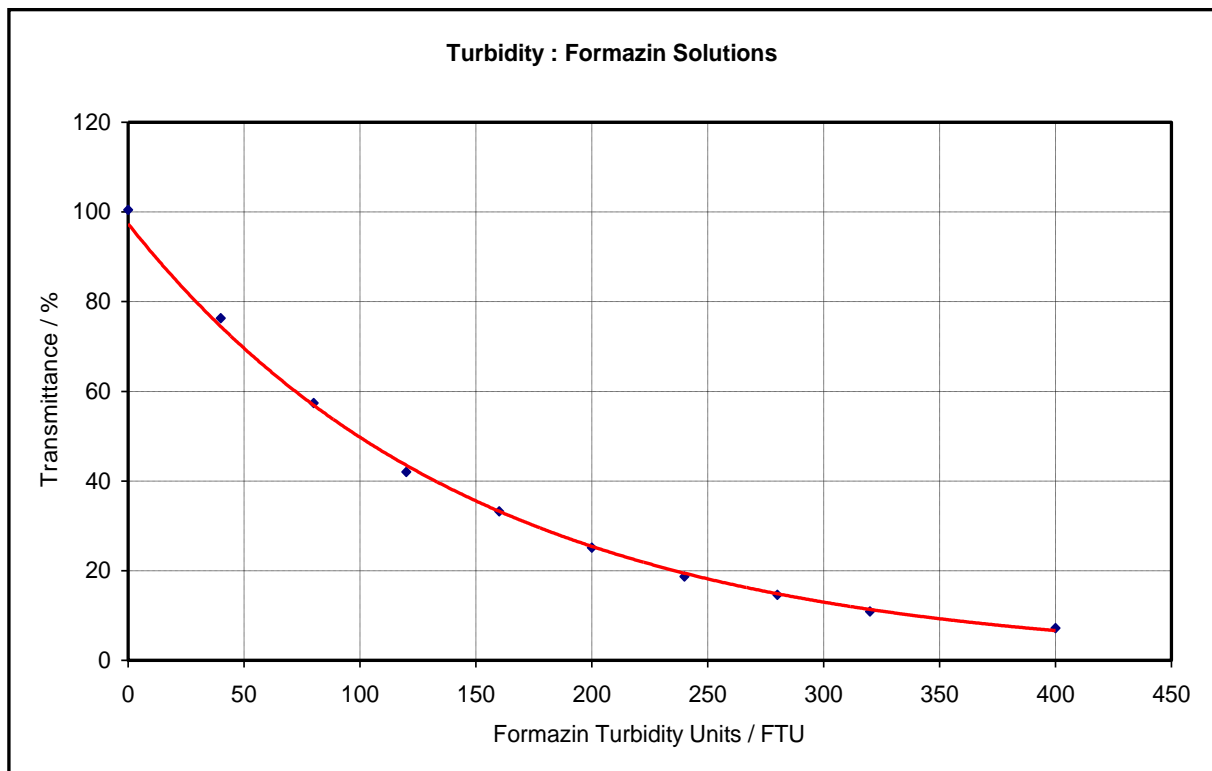
*Table 5: Sphericity & Roundness*

## 2.5 Turbidity

The following table and graph show the spectrophotometer calibration.

Solution/FTU	Transmittance/%
0	100.5
40	76.3
80	57.4
120	42.0
160	33.2
200	25.1
240	18.7
280	14.6
320	10.9
400	7.2

**Table 6: Turbidity Calibration**



Sample No.	Measured Sample Transmittance (%)	Turbidity (FTU)	Mean Turbidity (FTU)	Standard Deviation (FTU)
ISP (30/50)	80.5	32.5	30	4
	84.0	27.0		

**Table 7: Turbidity Results**

## 2.6 Crush Resistance

The result of the Crush Resistance testing is tabulated below.

Stress (psi)	ISP (30/50)		
	% fines, by mass	Average % fines, by mass	Standard Deviation (% fines, by mass)
10000	1.38	1.6	0.4
	1.91		
12500	2.65	3.1	0.6
	3.54		

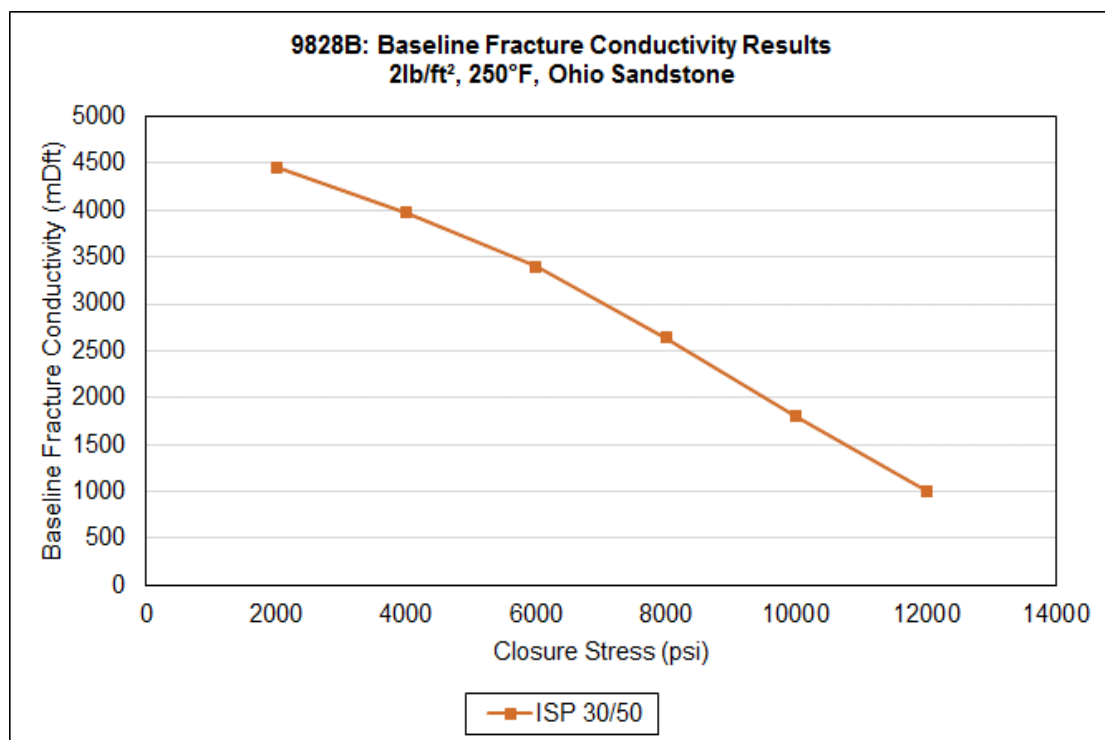
**Table 8: Crush Resistance Results**

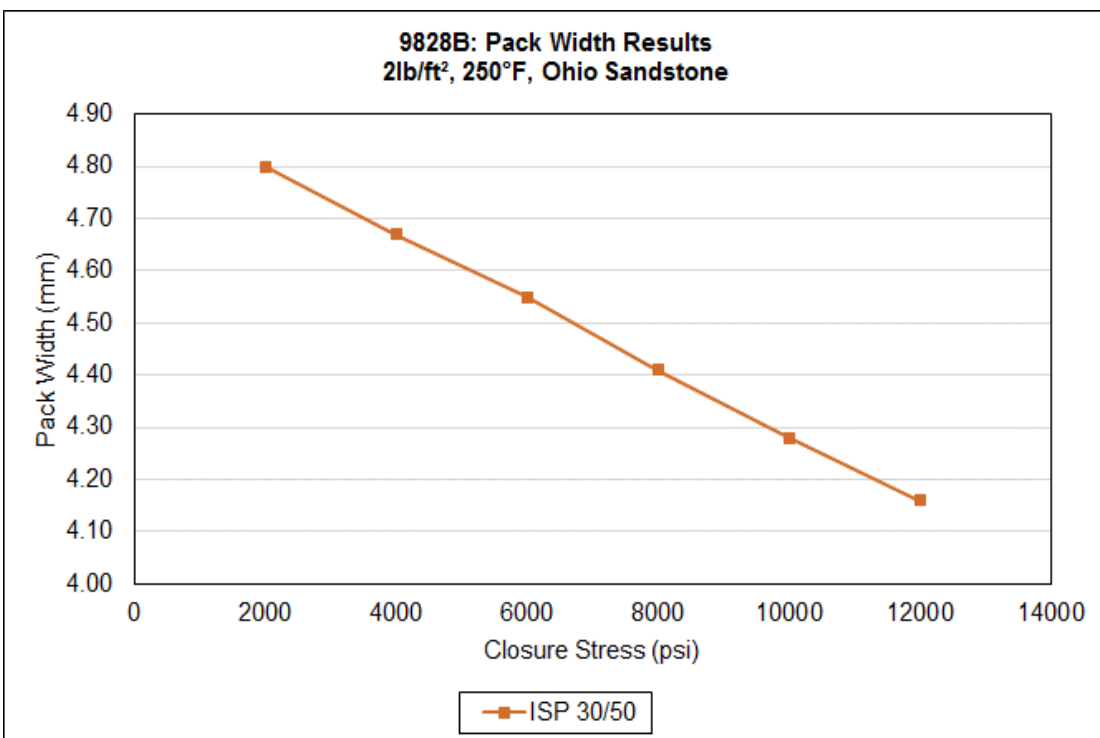
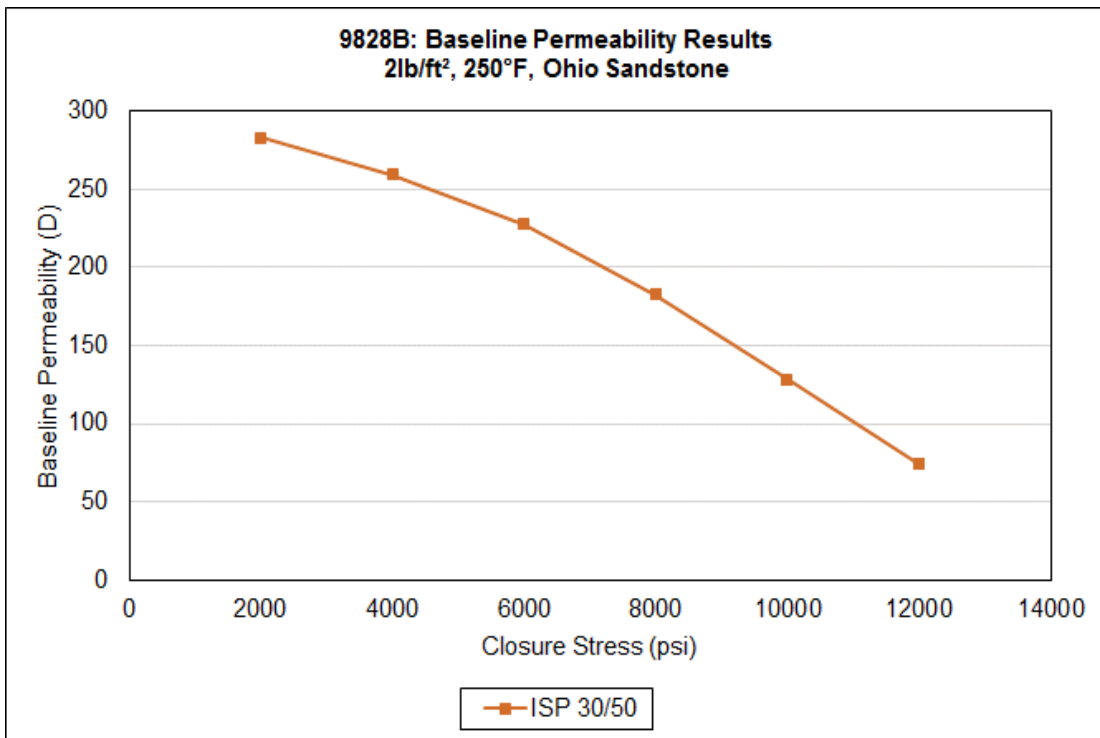
## 2.7 Baseline Fracture Conductivity & Permeability

The results of the baseline fracture conductivity; permeability and fracture widths are tabulated and plotted below.

Closure Stress (psi)	ISP (30/50)		
	Baseline Fracture Conductivity (mDft)	Baseline Permeability (Darcy)	Pack Width (mm)
2000	4456	283	4.80
4000	3971	259	4.67
6000	3396	227	4.55
8000	2642	183	4.41
10000	1799	128	4.28
12000	1010	74.0	4.16

**Table 9: Baseline Conductivity Results, 2lb/ft<sup>2</sup>, 250°F, Ohio Sandstone**

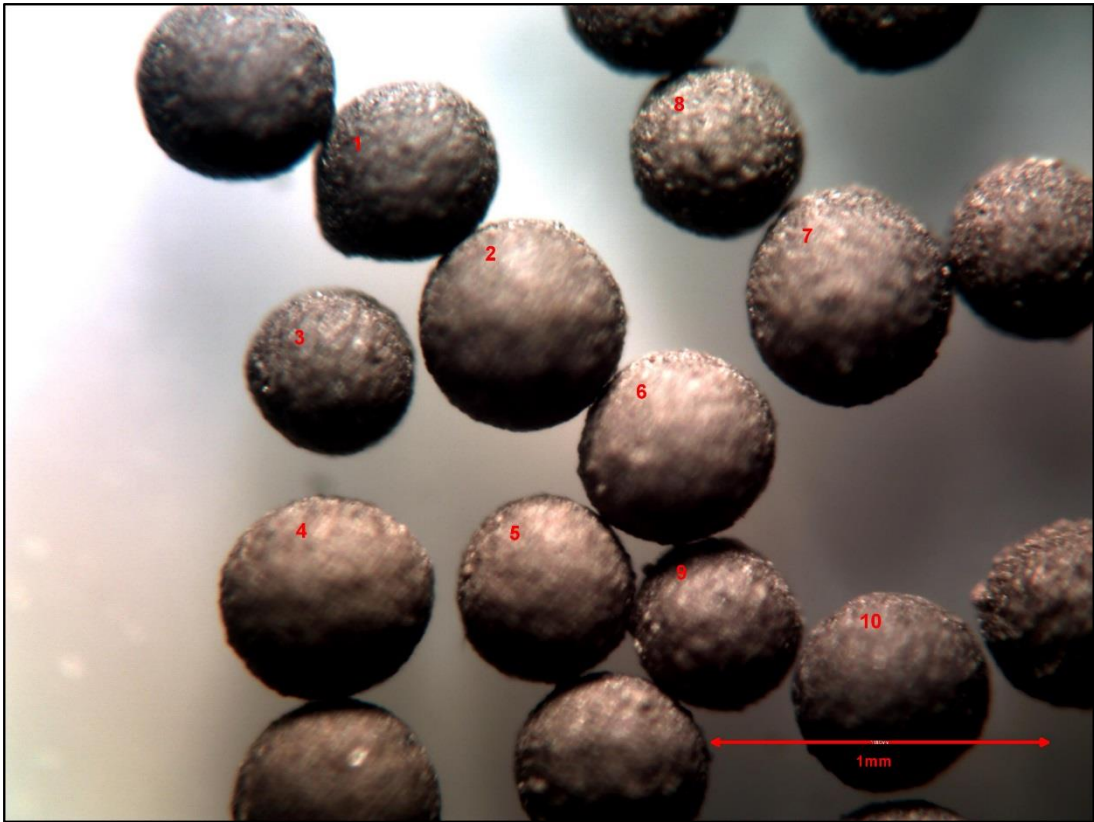




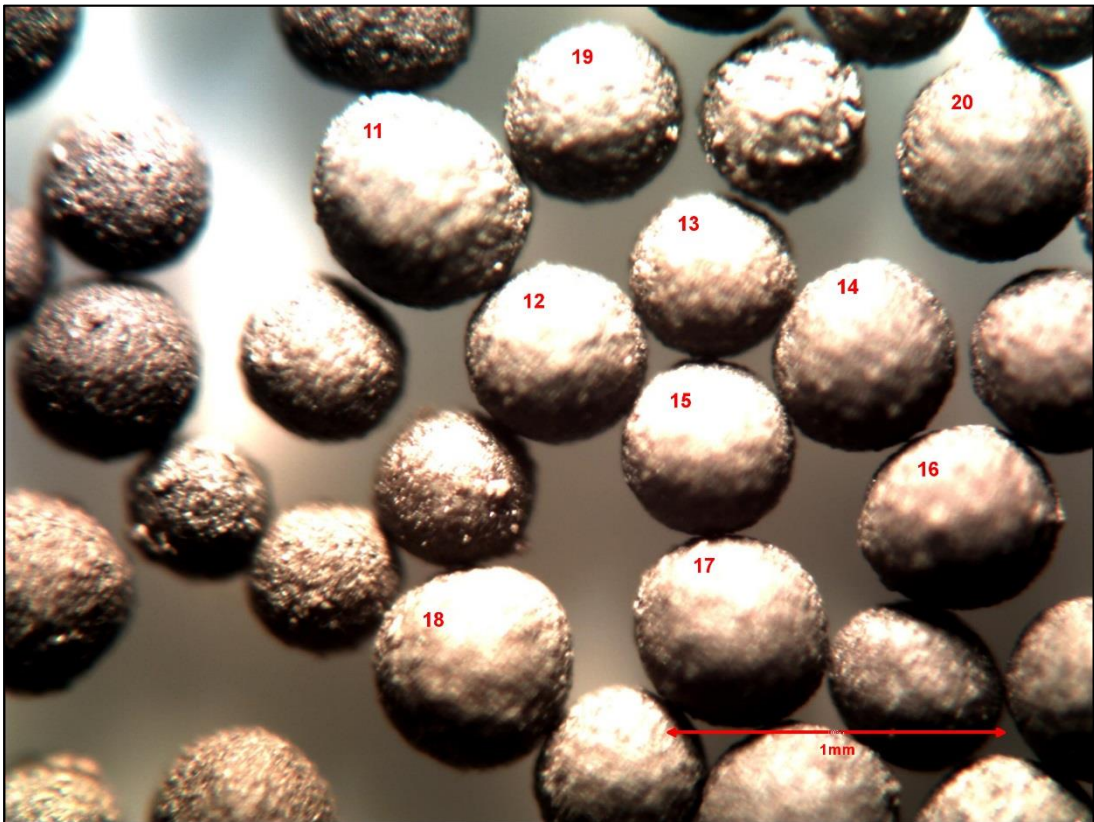


## **APPENDIX A**

### **Photomicrographs of test samples**



**Proppant Sample - ISP (30/50)**



**Proppant Sample - ISP (30/50)**