

# **Systematic Workflow for Characterizing Frac Sand: An Integrated Approach\***

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## **Abstract**

Unconventional and tight reservoir exploration and development activities in the Middle East region have increased and are expected to grow further. Developing such reservoirs has led to increased hydraulic fracturing activities; and consequently, increased demand for hydraulic fracturing sand. Hydraulic fracturing uses specially engineered fracturing fluids containing millimeter-sized grains of propping agents or silica sand to overcome stresses and keep induced fractures open to help ensure optimum hydrocarbon production. Sand used in hydraulic fracturing is aligned with quality standards outlined by the International Organization for Standardization (ISO) and the American Petroleum Institute (API).

The objective of this study is to develop a systematic workflow to characterize fracturing sand for hydraulic fracturing applications. This approach requires investigating the following aspects of sand used as proppants: 1) understanding of the mineralogical and elemental properties of sand; 2) analysis of geomechanical strength; and 3) determination of sand's suitability for use in fracturing applications. The ISO parameters testing is performed using X-ray diffraction (XRD) for mineralogical analysis and X-ray fluorescence (XRF) for elemental composition and other test methods as outlined in the ISO/API specifications for determining the bulk density, acid solubility, and turbidity. Sphericity and roundness of the particles are determined using a microscope. Also, crush tests are conducted for geomechanical characterization.

Existing dissimilarities in reservoir properties require an understanding of reservoir geomechanical and geochemical properties; hence, a fit-for-purpose characterization approach for using sand as a proppant. Planning is essential in unconventional reservoirs because the properties of each reservoir are unique and require customized approaches to design and develop a proper fracturing and proppant solution. This workflow should help well operators and service companies determine the type of proppants that suit the targeted reservoir formation, which should help optimize reservoir productivity.

## Introduction

Unconventional and tight reservoir exploration and development activities in the Middle East region have increased and are expected to grow further. Recently, the global market has been fed by hydrocarbon sourcing from deep unconventional reservoirs. Prevailing examples from the rest of the world include the Barnett, Haynesville, Woodford, Eagle Ford, Marcellus, and Fayetteville shales of North America (Rogner, 1997; Hill et al., 2004; Perry and Lee, 2007) and the Montney and Horn River formations in Canada. Such reservoirs have also been identified in Eastern Europe, Russia, China (Lu et al., 2012), Australia, and Saudi Arabia (Alexeyenko et al., 2013; Hayton et al., 2010). Developing such reservoirs (Pal et al., 2015) has led to increased hydraulic fracturing activities and, consequently, increased demand for hydraulic fracturing sand (Zoghbi et al., 2015). Hydraulic fracturing uses specially engineered fracturing fluid containing micrometer-sized grains of propping agent in the form of natural sand, coated sand, and/or man-made proppant to keep induced fractures open to help ensure optimum hydrocarbon production.

Further development to improve hydrocarbon production from unconventional wells involves a technique whereby pillars of fracturing sand surrounded by empty space are used to prop open the fractures as compared to filling the entire frac volume with sand or proppant. This is achieved by injection of sand/proppant in pulses to generate sand/proppant pillars using modified resins and epoxies to lock the pillars together, which play an important role in stable pillar formations. The concept behind pillar formations is to hold formation closure stresses and develop flow channels between pillars that results in extremely high conductivity.

Back in 1947, Arkansas River sand was used in hydraulic fracturing techniques as proppant (Montgomery and Smith, 2010). Three to four decades later, several new proppants were introduced in the market to fulfill petroleum industry demands. On a broad scale, proppant can easily be classified into the following classes: bauxites, ceramics, coated and uncoated sand. Bauxites and ceramics are considered as mechanically strongest among all types, providing high conductivity and durability and preserving petrophysical properties that ultimately lead to higher production (Gallagher, 2011). Whereas, resin-coated sand is considered as medium strength and natural (uncoated) sand is low strength, which corresponds to medium and low conductivity, respectively ([Figure 1](#)).

Synthetic proppant (bauxite ceramics) is widely used for deeper wells in which closure stresses are much higher. The chemistry of synthetic proppants is based on sintered bauxite, kaolinite, magnesium, silicate, and blends of bauxite and kaolinite (Liang et al., 2015). On the other hand, specially designed resins/epoxies are made to encapsulate natural sand grains to enhance mechanical strength. Furthermore, there are two practices followed by the petroleum industry. “On-the-fly” treatment coats epoxy or furan resins onto natural sand at the wellsite during fracturing treatments (Murphey and Totty, 1989; Underdown et al., 1980). In other practices, resins can be coated onto natural sand at a plant and can be fully cured or partially cured prior to the subsurface application (pre-coated sand).

The use of natural sand as the fracturing proppant has become more prevalent again during the last decade ([Figure 1](#)). High demand for natural sand is attributed to availability and cost effectiveness. The mined sand is subjected to crushing, cleaning/washing, removal of heavy minerals, and drying before it is distributed into appropriate sizes. Sand used in hydraulic fracturing is aligned with quality standards outlined by ISO and API.

This study focuses on developing a systematic workflow to characterize fracturing sand for hydraulic frac applications. Sand characterization is mainly divided into two main categories: 1) post-mining processes, and 2) qualifying parameters. Post-mining process is conducted after mining the sand from sand reserves. This process includes the removal of clays and heavy minerals, whereas the qualifying parameters are mainly based on ISO/API, which clearly states the benchmark for each of the parameters.

In this study, the authors shed some light on ISO/API parameters via microscale studies, including SEM-EDX and thin-sections. These additional key parameters are used to study surface morphology, micro-fractures, and pores. XRF and XRD are used to study elemental and mineralogical composition.

Four different sand samples were selected to compare and explore the reasons for different mechanical properties ([Figure 3](#)). The two samples from the Middle East (source 1 and source 2) were studied against the best available sand in the global market (source 3 and source 4).

### **Sample Preparation**

Hydraulic fracturing sand analysis starts with sample preparation, which is a basic but critical step. Figure 2 shows the split and reduced samples. The split and reduced sample best represents the entire pile or heap of sand either stored in sacks or trucks. Sample reducer reduces the size 1/16 times and is used to reduce the amount of sand. While, on other hand, splitter divides the sand into two equal halves to obtain appropriate size.

The four samples were received in 20-kg+ sacks, and all samples were individually reduced to a few kilograms using reducer. The quantity of reduced samples was further lessened by using splitter to acquire the needed sample.

### **Sieve Analysis**

Sieve analysis is a very important parameter in sand selection criteria. The concept behind sieve analysis is to restrict the grain sizes to a certain range (e.g., 20/40, 30/50, etc.). This helps with fracture job design and its successful application. ISO fracturing standards state that 90% of sand should fall in the designated sieves. [Figure 4](#) explains the sieve analysis of sources 1, 2, 3, and 4. Almost 90% of the studied sand sample falls between the specified sieves, and it passes the recommended criteria (API RP 61).

### **Morphology**

The morphology of sand grains are studied by two important parameters, which best explain the geometry of sand. Sphericity explains the spherical nature of grains while roundness covers the gentle edges of sand grains. Krumbien and Schloss (1963) visually explain the sphericity and roundness. ISO-acceptance range is greater than 0.6 for both sphericity and roundness ([Table 1](#) and [Figure 5](#)). Scanning electron microscope (SEM) works as dual-function equipment by providing surface morphology microphotographs and high-resolution images to view minerals, micro fractures, and micro pores (see [Figures 8](#) and [9](#)). Thin-section micro-images support SEM analysis by providing additional information in terms of crystallinity, morphology, and micro-fractures (see [Figures 8](#) and [9](#)).

Microscope images of sand grains ([Figure 2](#)) compared against Krumbein and Sloss (1963) ([Figure 5](#)) clearly shows all the sand passes the ISO/API criteria shown in [Table 2](#) (API RP 61).

### **Acid Solubility and Turbidity Tests**

Sand grains consist of varieties of minerals present in nature; the most common minerals are quartz, feldspar calcite, dolomite, etc. Of all these minerals, quartz is the most highly resistant to weathering. As per ISO/API criteria, under solubility test, sand or proppant is tested with 12:3 HCl:HF acids (i.e., 12% HCl by mass and 3% HF by mass). The acceptance range of solubility according to the ISO specification is less than 2%.

Turbidity is the optical property of particulate material while suspended in fluid; a high turbidity number corresponds to higher clay/dust size particles present in the fluid. In other words, turbidity is a measure of silt and clay size material suspended in fluid ([Table 2](#) and [Figure 6](#)). Reservoir petrophysical properties and frac fluid performance are sensitive to fines migration. Sand grains are sometimes contaminated with fines that are intact to sand grains. Turbidity of frac sand is measured by UV equipment. High concentrations of fines results in a higher turbidity number. Turbidity qualifying criteria is less than 250 FTU and/or NTU as shown in [Table 2](#).

### **Crush-Resistance Test**

Geomechanical properties of sand grains play an important role in keeping hydraulic fractures open. The key parameter is tested by crush test and supported by microstudies. Understanding the microstructures helps in interpreting the cross-comparison of crush test and permeability values. ISO/API reports 10% fines generation as the maximum limit for sand application at the specified formation closure stress. Sample 1 and 2 fail at 4,000 psi and are considered as comparatively weaker sand, whereas sample 3 generates 10% fines and lies on boundary line. Sample 4 possesses higher compressive strength compared to samples 1, 2, and 3 Z ([Table 3](#)).

### **Mineralogy**

XRD analysis determines the minerals present in the sand. Sand samples are crushed, passed through 200  $\mu\text{m}$  sieve, and run under XRD. The XRD spectrum covers a wide range from  $4\theta$  to  $80\theta$ . Energy-dispersive XRF is used to study the elemental composition of minerals present in the sand samples, and it assists in selecting the right minerals during XRD analysis. Both advanced techniques (XRD and XRF) reveal the dominant composition of silica ( $\text{SiO}_2$ ), ranging more than 90%. Apart from silica, feldspar and traces of pyrite is observed.

### **Discussion**

All possible factors covering characterization of fracturing sand including geomechanical, geochemical, and physical analysis should be tested prior to qualifying the material for use in fracturing applications. A sand characterization flowchart which provides a view of this testing is provided in [Figure 7](#). High-resolution microstudies ([Figures 8](#) and [9](#)) are also helpful in determining the viability of the sand for fracturing

applications. The high-resolution microstudies provide additional information that is interlinked with the properties analyzed in the typical ISO tests. This helps in understanding the hydraulic fracturing sand from almost all perspectives.

At first glance, all four samples appear to be feasible for subsurface application since these samples pass almost all ISO/API parameters. Sieve analysis shows more than 90% value falling between the 20/40 mesh size. The rest of the parameters, such as morphology (sphericity and roundness), turbidity, and solubility also successfully pass the ISO/API parameters (API RP 61). The geochemical analysis also reveals the maximum composition of quartz, which is supported by XRF results.

Geomechanical properties after crush-resistance testing show source 4 sustains 5,000-psi closure stresses (9.5% fines), source 3 sustains 4,000 psi (10% fines), and source 1 and source 2 sustain closure stresses less than 4,000 psi ([Table 3](#)).

The high-resolution photomicrographs disclose the reason for the different sand behaviors and their response to mechanical stress. SEM and thin-section photomicrographs reveal sources 1 and 2 are highly fractured/porous and medium rounded. Fracture lengths range from 200  $\mu\text{m}$  to 500  $\mu\text{m}$  in size and vary from sample to sample. [Figure 8](#) reveals the presence of 10 to 30- $\mu\text{m}$  sized micro pores in most grains of source 1 and source 2. Such features indicate these sand grains were subjected to regional tectonic stresses, overburden, and dissolutions.

SEM and thin-section images reveal relatively medium maturity level of source 3 and high maturity level of source 4 ([Figures 8](#) and [9](#)). Sand grains in sources 3 and 4 are fracture-less, smooth, well rounded, and spherical ([Figure 9](#)).

Mineralogical, sedimentological, lithofacies, and other parameters of a sandstone reservoir are controlled by environment of deposition, provenance of sediment, diagenesis, compaction dissolution, recrystallization, and structural deformation (from micro to megascale). These key controlling factors directly affect the individual sand grains. Morphological parameters are controlled by deposition environment of provenance rock and type as well as travel distance. Sediments/sand grains sourcing from quartz-rich geological formations travelling long distances and deposited in high-energy environments result in well-sorted, well-rounded, and well-spherical geometrical structure. When these grains/sediments are buried into the subsurface, diagenesis with the involvement of temperature and pressure (compaction) modify the geometrical properties of sand grains and help enhance mechanical properties in terms of maturity.

## Conclusion

Systematic evaluation of fracturing sand involves tests, such as sieve analysis, solubility, turbidity, mineralogy, and crush-resistance (ISO/API). Coupling this information with high-resolution microphotographs provides additional information that helps explain some of the ISO test results. High-resolution microphotographs determine the intrinsic reason for weaker sand \_sand is attributed to surface morphology, micro fractures, and micro pores. Sources 3 and 4 are considered the best market sand, which is also revealed in SEM and thin-sections images. Whereas, surface undulations, micro fractures, and micro pores (defects), which generate a higher percentage of fines at certain stress levels, are prominent in sources 1 and 2. Such types of sand generate a high percentage of fines under reservoir conditions; ultimately, damaging reservoir petrophysical properties. However, these defects if treated with resins to fill fractures and pores and encapsulate the grain size can eventually improve mechanical strength. Consequently, this integrated approach is highly recommended to understand ISO parameters and

benefit decision makers through better screen sand samples.

### **Acknowledgments**

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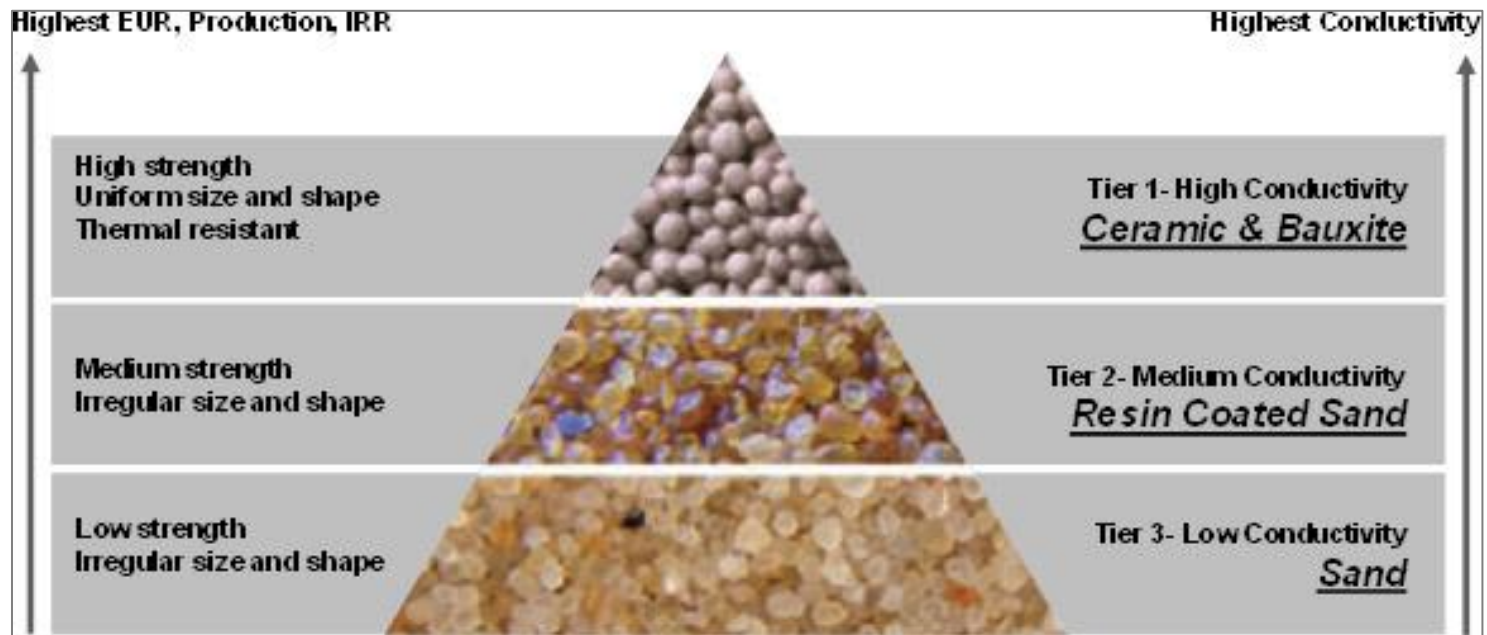


Figure 1: Distribution of sand according to mechanical strength and conductivity (modified after Gallagher 2011).



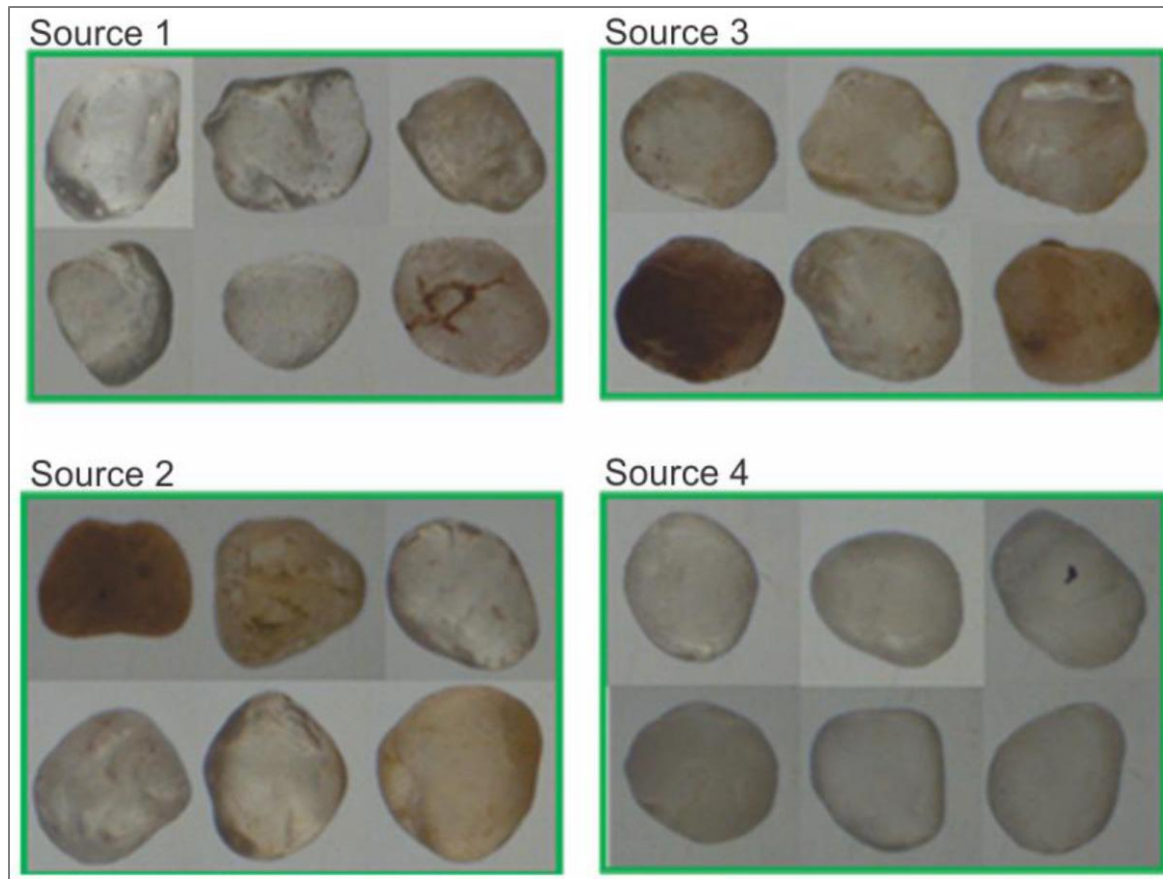


Figure 2: Sand samples for study.



Reducer



Splitter

Figure 3: Sample preparation using splitter and reducer.

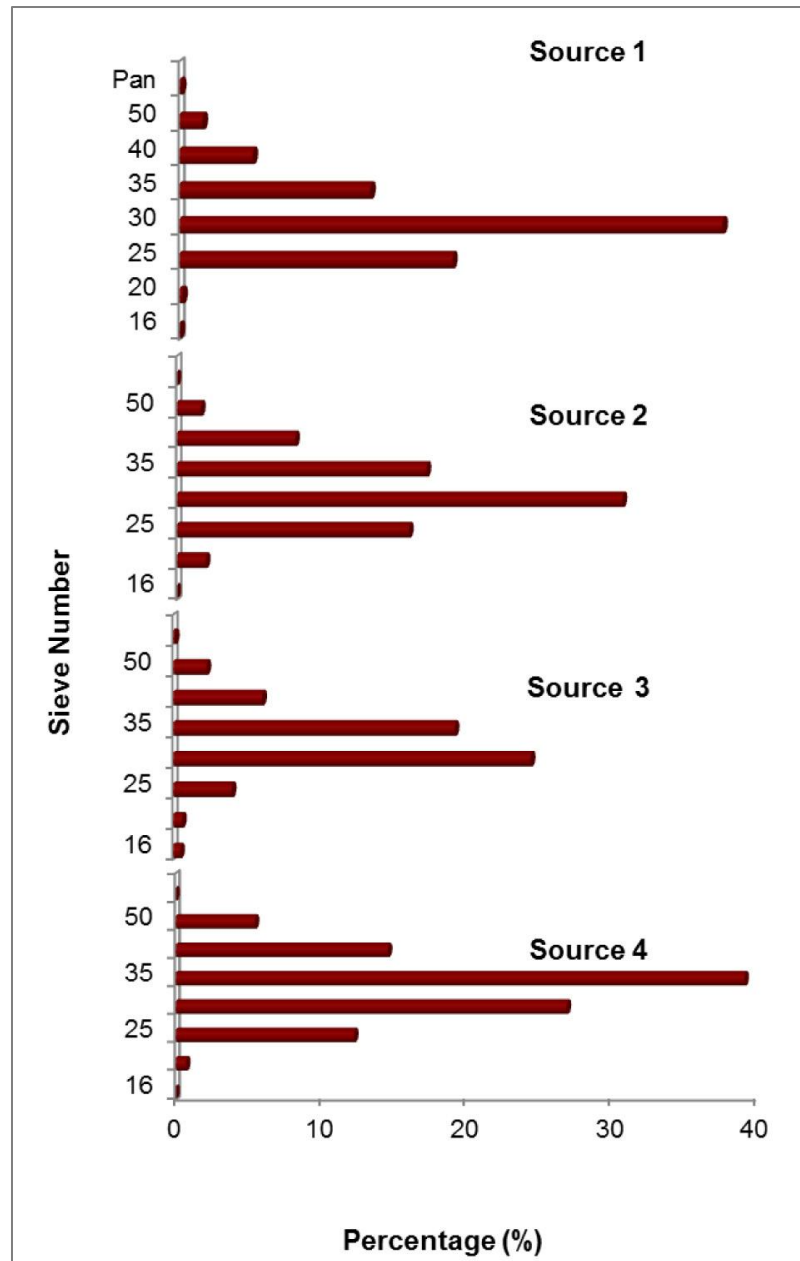


Figure 4: Sieve analysis for source 1, 2, 3, and 4 samples. All four samples contain more than 90% sand between the designated slides.

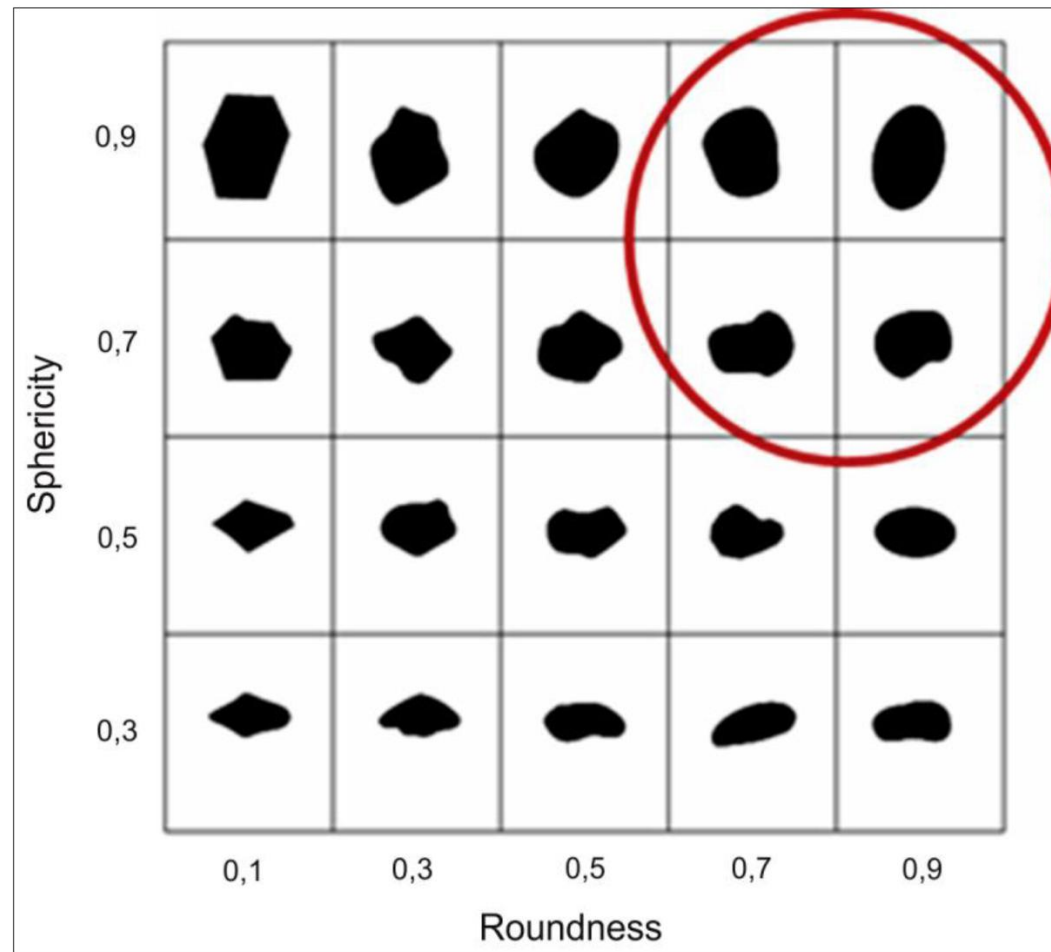


Figure 5: Krumbein/Sloss' (1963) visual estimate chart of roundness and sphericity.



Figure 6: Turbidity comparison of clean sand sample versus clay and silt-bearing sand sample.

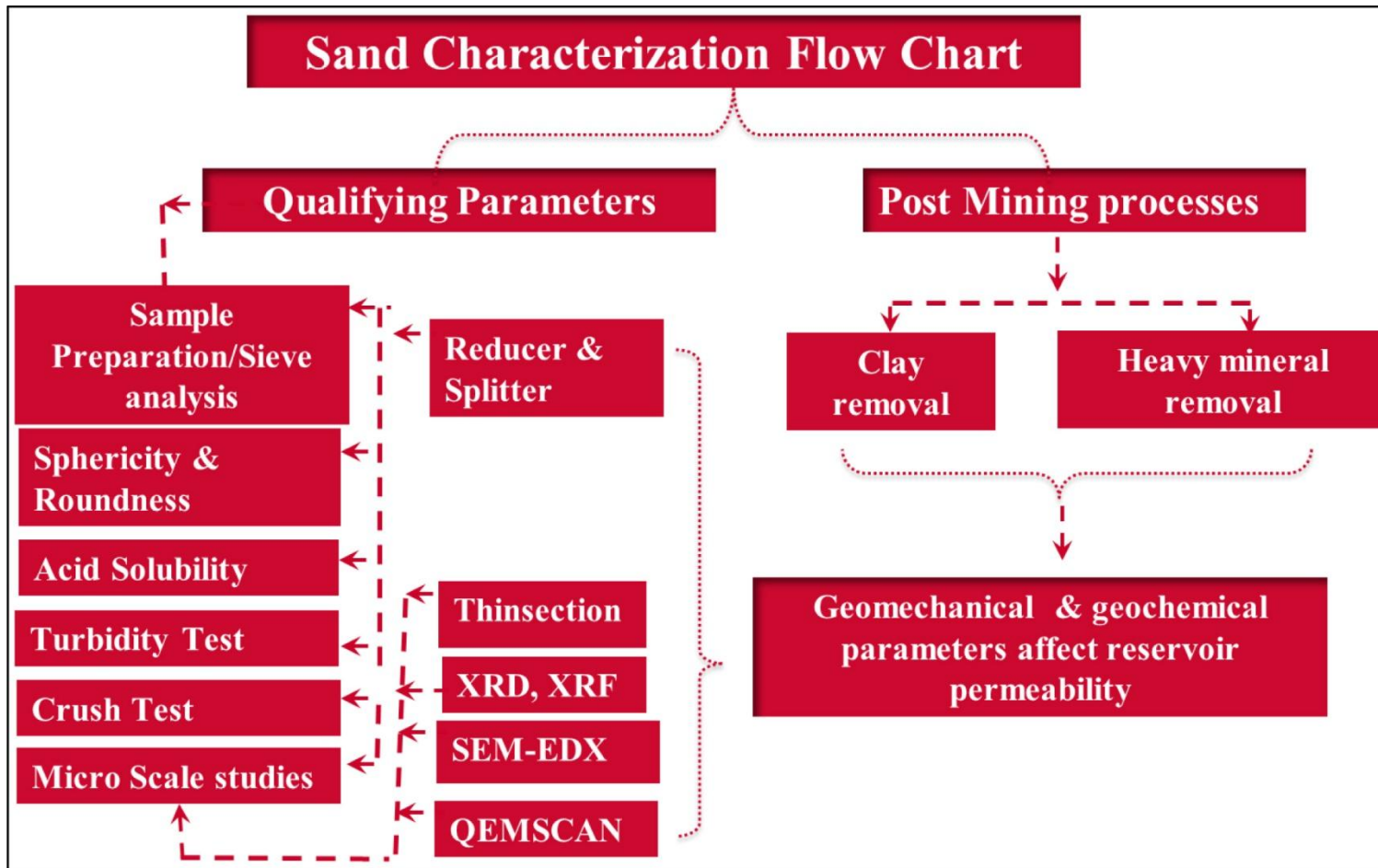


Figure 7: Detailed workflow to characterize fracturing sand.

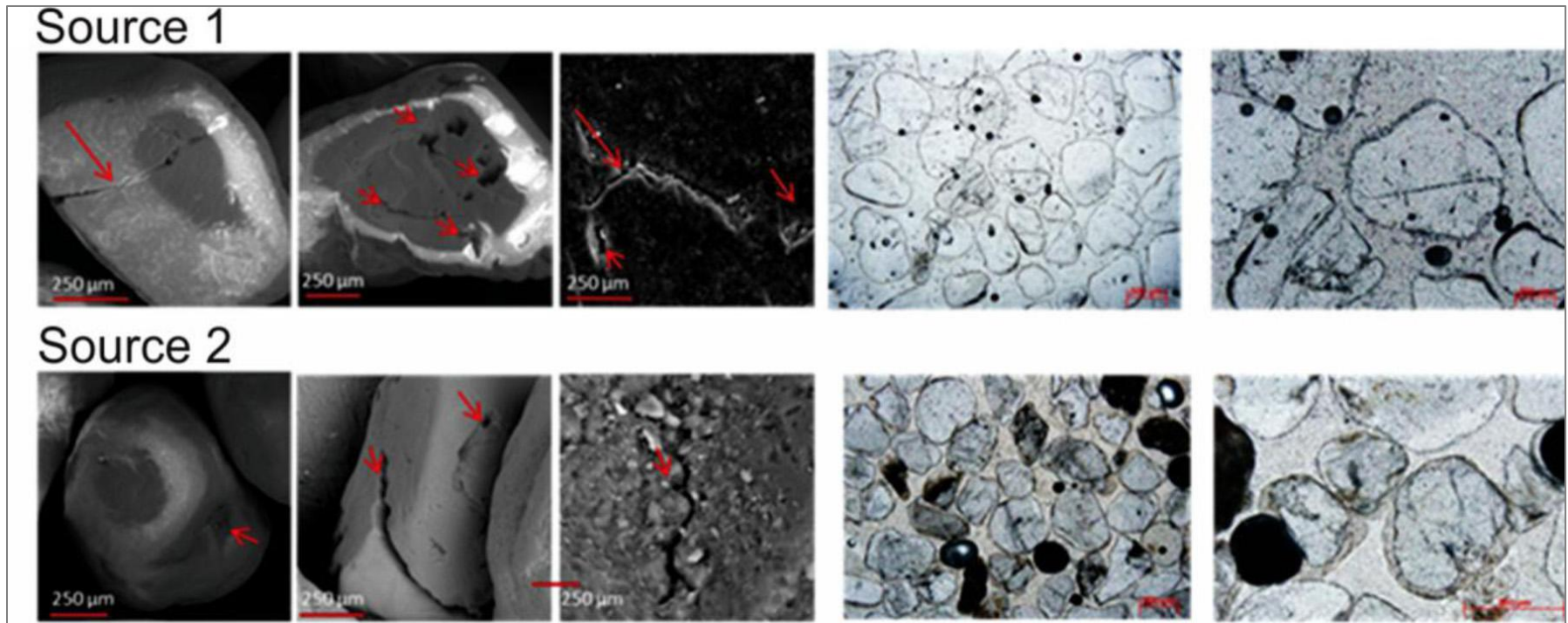
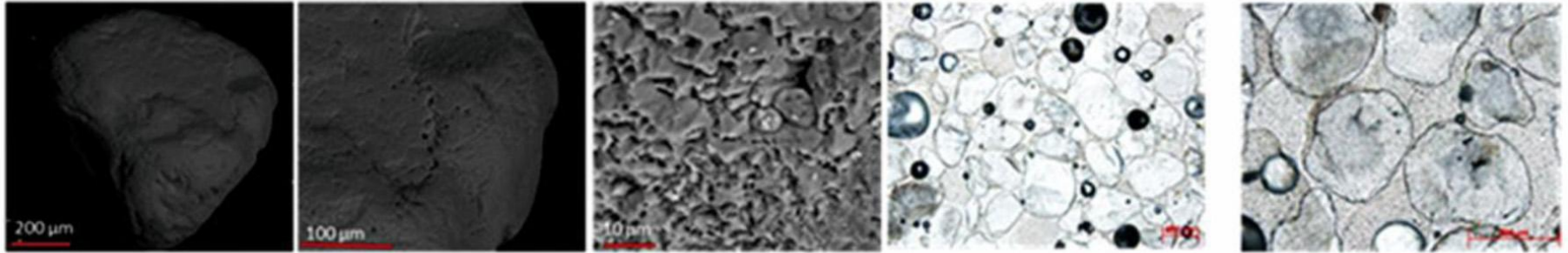


Figure 8: SEM image (left) and thin-section (right) of source 1 and source 2 sand samples showing micro fractures and micro pores on the surfaces.



## Source 3



## Source 4

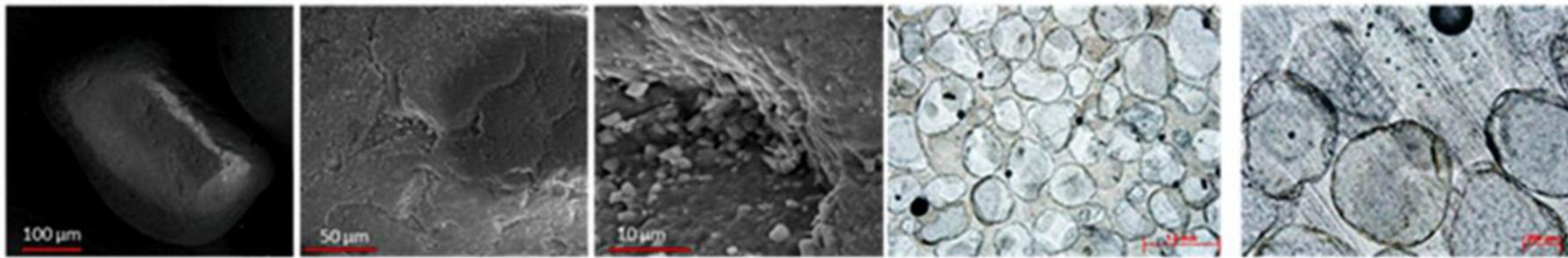


Figure 9: SEM image (left) and thin-section (right) of source 3 and source 4 showing no evident micro fractures or other imperfections.

Test		Natural Sand 20/40				
		Source 1	Source 2	Source 3	Source 4	ISO/API
Morphology	Sphericity	0.765	0.74	0.7	0.8	+0.6
	Roundness	0.705	0.7	0.7	0.8	

Table 1: Sphericity and roundness of 20/40-sized selected sand.

Test	Natural Sand 20/40				
	Source 1	Source 2	Source 3	Source 4	ISO/API
Solubility (%)	2.4	2.67	1.6	1.2	2% maximum
Turbidity (FTU)	50	65	18	20	250 NTU maximum

Table 2: Solubility and turbidity results.

Test		Natural Sand 20/40				
		Source 1	Source 2	Source 3	Source 4	ISO/API
Crush Strength (% fine)	2,000 psi	2.08	2.34	–	0.38	10% maximum at any stress load
	3,000 psi	6.2	4.89	4.8	1.02	
	4,000 psi	14.4	11.6	10	2.78	
	5,000 psi	–	–	22	9.5	

Table 3: Geomechanical properties of natural sand.