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# Characterization of a new polymer-nanocomposite proppant from agro-waste products for hydraulic fracturing operations

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**Abstract**

Hydraulic fracturing is the main operation to stimulate wells and starts with pumping fracturing fluids into the well to raise bottom hole pressure above formation fracturing pressure. Once a fracture is induced, a slurry with proppant is injected to keep flow path open for reservoir fluids towards the wellbore at a higher rate. Due to the high expense of conventional proppant types that reaches up to 40% of the stimulation job, the need for new proppants has become a very important topic of research. This research comprises an experimental study to characterize a proppant prepared from Polymer Nano-composite materials added to the rice husk to act as a possible propping agent. The physical and mechanical properties are investigated, and a fracture conductivity test is implemented to characterize the efficiency of the new proppant material. The experimental results are compared to the commonly known walnut hull proppant (ULW-1.25) and Chemically Modified and Reinforced Composite Proppant (CMRCP). The new polymer Nano-composite proppant showed promising results according to the established ISO/API standards. These results may lead to a consequent improvement towards high strength Nano-composite proppants for applications in hydraulic fracturing operations and other petroleum engineering applications.

***Keywords:*** A new polymer; Nano-composite proppant; hydraulic fracturing; well stimulation; fracture conductivity test.

## 1. Introduction

Viral Hydraulic fracturing is an inexpensive technique that has been used since 1949 in the petroleum industry to improve oil and gas production [1]. It involves generating highly conductive fractures into tight petroleum reservoirs to recover hydrocarbons through these induced channels and improve the overall reservoir productivity [2]. The hydraulic fracturing method uses fluids to create fractures within a formation by increasing the applied pressure to overcome rock strength [3]. This results in the creation and propagation of the fractures which enhances the productivity of the formation [4]. This stimulation method consists of four stages; fracture initiation, fracture propagation into the hydrocarbon bearing zone, maintaining the induced fractures opened against the formation closure pressure, and finally achieving a suitable drawdown to evaluate its effect on the rate of production [5]. This requires evaluation of the reservoir properties [6-8], geomechanical properties [9], and wellbore stability in the stimulated interval to keep fracture propagation within the target zone [10]. Optimized stimulation operation typically deploys mechanical earth models [11] and hydraulic flow units' analysis [12] to help design the proper placing of fracture sets [13]. To perform this treatment, various types of fluids are needed. However, fracturing fluids are not only playing an important role in initiating and/or propagating the induced fractures, but also maintain these fractures open by transporting and inserting a suitable propping agent to settle against fracture walls [3]. These activities usually entail uncertainty and must be handled carefully to avoid repercussions of failure [14, 15].

This propping agent may be aluminum balls, sand, glass beads, walnut-hulls, ceramics, plastic balls, or resin coated proppant [16]. Currently researchers are working on enhancing the quality of proppants to eliminate their defects for using in hydraulic fracturing treatments [17]. For example, the use of sand as a proppant which has an average specific gravity of 2.5 to 2.69 is very common in hydraulic fracturing treatment due to its low cost and ease of access. However, the main disadvantage of proppant sand is the lack of enough strength to withstand against confining crushing pressure [18, 19]. When the frac sand suffers crushing, small fines are produced and may cause plugging the induced fracture or proppant flowback, decreasing fracture conductivity, erosion of the piping and facilities, and reduction in well productivity [20]. Recently, modern techniques for reservoir fracturing have been suggested [21, 22] to enhance the oil recovery and prevent reservoir

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deterioration while drilling [23, 24].

Modern Technology in reservoir fracturing involves the development of novel approaches to introduce new proppants, structure stabilizer, dye-tracers, and degradable fiber slickwater. Rafał Morga [25] presented producing ultra-light weight coke proppants for fracturing deposits of coal bed methane from foundry coke, blast-furnace coke and a coke breeze. Experimental characterization of the new proppant mainly showed macroporous materials of porosity up to 40 % with cylindrical, less frequently bottle-shaped or wedge-shaped pores. The new coke proppants met all the requirements of the ISO 13503-2:2006/Amd.1:2009 standard except for crush resistance. Peipeng Yang et al. [26] provided a novel approach to effectively utilize secondary aluminum ash, a solid waste, with kaolin as a binder to entirely replace the main raw material of bauxite in preparing Ceramsite proppants. The new product is fully described by SEM images and the results are subsequently supported by lab measurements including bulk density, apparent density, acid solubility, and crush rate. Jiang Guo et al., [27] tried to modify the properties of quartz sand proppant surface to excellent oleophilic and hydrophobic using long-chain fatty acid (lauric acid, myristate acid or stearic acid) and nanotantia (TiO2) composites (LATC, MATC and SATC). Experimental results indicated that the SATC@sand proppant showed better thermal stability with thermal decomposition temperature of about 170°C. Meanwhile, it revealed a relatively lower acid solubility, apparent density and bulk density compared to the conventional sand proppant, indicating great potential for use as a proppant in hydraulic fracturing. Ricardo Anaya et al., [28] assessed the feasibility of reusing significant green glass from urban waste with commercial red clay, potassium and sodium feldspars to produce low-density ceramic proppants. The resulting granules are characterized using the international proppants standard with scanning electron microscopy, X-ray diffraction, and individual diametral compression tests. Experimental results revealed competitive proppants with low-density and good breakage ratio. Jianchun Guo [29] developed a novel structure stabilizer (SS) to facilitate the formation of strong fiber-proppant agglomerates using fiber, polymer, and quartz sand. These agglomerates exhibit lower density, larger volume, and high contact area with the fluid during settlement compared to plain proppants. Such conditions typically improve buoyancy and drag forces that enable slow settling velocities and enhance deep transportation into fracture. Experimental lab results specified the optimal mass fraction of SS as 0.3% and application in over 80 wells in shale oil, shale gas, and tight gas reservoirs validated general suitability and strong adaptability for enhancing production, sand control and cost reduction. Min Ren et al. [30] optimized the preparation of multi-colored dye-tracer proppants for quantitative localization and volume assessment of proppant flowback in multistage fractured horizontal wells. Results showed that all the eight dyes varieties can effectively and evenly dye quartz sand proppant at surfactant concentrations of 0–200 mg/L and pH values of 6–13. Alternatively, temperature showed a negative effect on stain stability while fracturing fluid and salinity did not affect its stability. These dye tracers provide a new technique to monitor sand production after fracturing in horizontal wells. Such a monitoring technique provides valuable data to improve the efficiency of subsequent fracturing operation in unconventional reservoirs. Mingwei Zhao et al. [31] innovatively developed a degradable fiber slickwater fracturing fluid system to enhance proppants-carrying mechanism. Several lab experiments investigated the rheology of the fiber slickwater together with the static and dynamic proppants-carrying experiments to evaluate the proppants-carrying performance. Results revealed significantly improvement in viscoelasticity of fiber slickwater than pure slickwater, stronger structure, and 20–1000 times enhancement in zero-shear viscosity. In addition, results of the dynamic proppants-carrying experiments showed dramatic reduction of proppants residue near-wellbore with uniform proppant placement and About 11.8% proppants delivery to the fracture distal end. A LMPS fracturing fluid [32] comprises a self-assembled low molecular weight polymer and a surfactant that can establish short robust molecular networks among many self-assembling association sites. It demonstrates strong elastic characteristics with a minimal loss tangent in the viscoelastic response as low as 0.1. During the fracturing process, it possesses superior capability towards proppant suspension, and stronger resistance to deformation, thereby preventing proppant settling. Finally, in-situ generated proppants (IGPs) present new proppant generations that avoid the disadvantages of pre-made proppants. IGPs are based on liquid-to-solid phase transformation under high-temperature and high-pressure available at typical reservoir's conditions. Such transformation may initiate by chemical reactions, changes in temperature, pH, … or other conditions, and inconsequence the fracturing fluid is transformed in situ into strong solid and heat-resistant proppants. Mohan Raj Krishnan et al. [33] summarized in a comprehensive review the recent advancements in preparing and properties of (IGPs) for an effective hydraulic fracturing operation.

As a result of progress occurred in proppants field over the past seven decades, the proppant market is predicted to utilize new materials. Although the conventional types of proppants (for example, frac sand, ceramics, RCP, etc.) have been used in hydraulic fracturing treatments for many years, they still suffer from a significant weakness as fracturing fluids should have enough viscosity to transfer the proppant particles inside the induced fractures. New methods aim at using high-strength and low-density substrates (i.e. deformable substrates) to achieve operational and economic goals [34]. Many efforts have been exerted to develop new strong propping agent materials of light weight. The effectiveness of using nutshells, like walnut hulls, was investigated in 1960 [35]. While the walnut hull substrate possessed high strength and lightweight, it is reasonably deformed, rather than crushed, when subjected to high formation closure pressure. A larger exposed area against the reservoir formation wall is the result of this deformation, which contributes to stress reduction. Another feature of this material is dispersibility and ability to distribute property within the created fracture that leads to better flow capacity [36]. Correspondingly, utilizing the Ultra-Lightweight (ULW) proppants has increased quickly. ULW proppants have great ability to be used with very low viscous liquids [37]. A propping agent with a specific gravity of less than 2.0 can be considered as an ULW proppant. It is generally accepted that utilizing ULW proppants can contribute to increasing the flow capacity of induced fractures and in consequence improve hydrocarbon recovery [38]. In the present study, the main physical and mechanical properties of a new Polymer-Nanocomposite Proppant from Agro-waste products are characterized to assure suitability for hydraulic fracturing operations. The tested physical properties of a selected sample involve grain size distribution, grains shape, bulk density, and specific gravity. Turbidity and acid solubility tests, typical for evaluating mechanical properties of the new proppant, are also conducted. In addition, a fracture conductivity test is conducted in laboratory to simulate a propped fracture in reservoir conditions and evaluate the efficiency of the new material as a proppant. Finally, all lab results and measurements are compared to the corresponding characteristics of other proppant types presented in the literature such as walnut hull proppant (ULW-1.25) and the Chemically Modified and Reinforced Composite Proppant (CMRCP).

## 1. Test methodology

The new proppant product can be described as Polymer Nano-composite proppant based on agro-waste of rice husk to act as a propping agent in hydraulic fracturing treatment. The new polymer Nano-composite proppant is created in a multi-step process. During the first step, the row rice husk biomass was crushed and milled using the Ball Milling machine. Then this crushed rice husk was sieved in different sizes using Sieve Shaker machine. A powder filler of polymer nanomaterials with a size less than 100 nm, representing 80% of the total mix, was added to 20% by the weight of this crushed rice husk. Finally, the proppant was cut into small particles with size less than 450 microns to give the final product. The purpose of this study is to characterize the properties of the new polymer Nano-composite proppant for potential use as a new proppant in hydraulic fracturing treatment according to the recommended testing methodologies for evaluation and testing new proppant materials of API RP 56 (the American Petroleum Institute), and ISO (the International Organization for Standards). The tests performed in this research involve:

1. Testing the physical properties of the new proppant by determining the grain size distribution through sieve analysis of a selected sample, describing the grains shape using microscope, measuring the bulk density, and determining the specific gravity [26].
2. Testing the mechanical properties of the proppant by conducting the turbidity and acid solubility tests [28].
3. Performing the fracture conductivity test and comparing the obtained results with other proppant types presented in the literature.

### *1.1. Testing of the physical properties*

#### Sample preparation and grain size analysis

A sample of the new polymer Nano-composite proppant was cleaned, dried and sieved by a sieving tool. The sample was put in a set of sieves which are arranged in descending order and shaken for 0.25 hour according to ISO 13503-2-6 recommended time to obtain full classification of the grains. These proppant particles must meet the ISO requirement for more than 90 percent of proppant particles falling between two sieve screens.

#### The grain shape

For the shape description of proppant particles, random samples were selected and examined by the optical microscope for each sample as recommended by ISO 13503-2-7. The microscopic images for each sample were snapped and the appropriate shape of the grains was verified. Figure 1 shows an image of the selected sample for the examination.

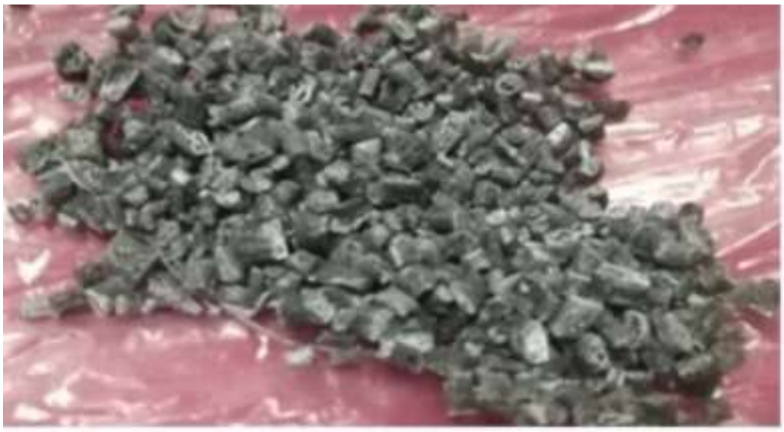

Fig. 1: The selected sample for the examination.

#### The bulk density

The bulk density involves a dry test to obtain the weight of the new proppant that fills a unit volume, which includes both proppant particles and the pore spaces. The bulk density is estimated by calculating the volume occupied by a pre-weighted sample and this step is repeated many times for accuracy as per ISO 13503-2-10 recommendation. An empty 100 ml cylinder is put on a weighing machine and the machine reading was adjusted to zero. After that, the measuring cylinder was filled with proppant samples until the reading reached 100 ml. The reading was recorded, and the bulk density was measured from Equation (1).

$$Bulk\ density = \frac{Weight\ of\ dry\ samples(g)}{Volume\ of\ dry\ samples(ml)} \quad (1)$$

#### The specific gravity

Specific gravity is determined by the ratio of the density of a substance to the density of a given reference substance. The density of proppant was determined by recording the volume of water that is displaced by pre-weighted amount of the proppant. According to API recommendations, kerosene with average density of 0.795 g/cm3 was used as a reference substance to measure the specific gravity of the proppant. Once the density of the selected sample was obtained, specific gravity is calculated according to Equation (2).

$$Specific\ Gravity = \frac{Density\ of\ proppant\ (g/cm^3)}{Density\ of\ kerosene\ (g/cm^3)} \quad (2)$$

*1.2. Testing the mechanical properties*

**The turbidity test**

ISO 13503-2-9 defines turbidity as the number of suspended grains or other divided materials present in a fluid substance. Turbidity test determines the amount of light that will pass through a fluid that is used as a wetting phase. A higher measurement indicates more suspended particles are present. Turbidity is measured by putting 1 gram of proppant in 25 ml of water and measurements are conducted using “HACH 2100N TURBIDIMETER”, Figure 2. The unit of measurement is Nephelometric Turbidity Units (NTU) and higher NTU measurements typically indicate inappropriate proppant handling and / or manufacturing practices.

**The acid solubility test**

The acid solubility test is considered an indication of the quantity of unwanted impurities present in the substance used as a propping agent. Exposing the proppant to acids may dissolve a part of the proppant material, deteriorate the propping capabilities, and decrease the conductivity of the created fracture in the area in-contact with these acids. The reduction of fracture conductivity near the pay zone may lead to a significant decrease in the well productivity. ISO 13503-2 recommendations for proppant acid solubility standardizes the test by using 12% by weight of hydrochloric acid (HCl) and 3% by weight of hydrofluoric acid (HF) as an indication of the soluble contaminants presented. This test was conducted to evaluate the suitability of the proppant for use in oil fields where the proppant may come into direct contact with acids. In the first step a 12:3% HCl- HF solution was prepared. To prepare 100 ml of HCl-HF 12:3%, a graduated empty cylinder was filled to 85 ml of distilled water and then 12 ml of HCL was added. After that, 3 ml of HF was added to the solution and stirred to confirm complete mixing. In the second step, a 5-gram sample of the proppant was weighted and added to the 100 ml solution, prepared in the previous step. This test was conducted at 150ºF for 30 minutes (ISO 13503-2). Finally, the proppant sample was dried in an oven for 20 hours and its final weight was measured, and the acid solubility evaluation is quantified using Equation (3).

$$The\ acid\ solubility(\%) = \frac{Propp.wt.before\ test - Propp.wt\ after\ test}{Propp.wt.before\ test} x100 \quad (3)$$

*1.3. The fracture conductivity test*

The fracture conductivity is an important test in this research because it indicates proppant suitability as a propping agent in the hydraulic fracturing treatment. Fracture conductivity test has been conducted according to ISO 13503-5. All tests were performed at 176ºF using 2% KCl solution as a fracturing fluid. The conductivity test was done utilizing fracture conductivity model especially designed for this experiment. This conductivity cell can work under a high closure pressure of 7500 psi and a temperature of 350°F. Figure 3 shows the fracture conductivity cell that includes a constant confining pressure pump, constant flow pump, core holder and back pressure regulator. Oligocene sandstone (from Cairo - Elsokhna desert road) core sample platens (Figure 4) were utilized as a common reservoir rock representative. Figure 5 shows a schematic illustration for the laboratory setup for conducting fracture conductivity test. The proppant sample was prepared and sandwiched in-between the two Oligocene sandstone platens as shown in Figure 6.

Based on the ISO 13503-5 procedures for measuring proppant conductivity, the following criteria are considered during the experiment:

1. The back pressure regulator should be able to maintain a pressure difference of 300 to 500 psi between the applied pressure (injection pressure) and the closure pressure (over-burden pressure). For example, if the injection pressure is 200 psi, then the applied closure stress shall be 500 psi greater (i.e. 700 psi).
2. All ramp rates should be at 100 psi/min.
3. The test core holder temperature shall be maintained at (176ºF).
4. To ensure the data points are in a statistical range, at least five data points should be recorded, and the average proppant pack conductivity shall be calculated over a range of 2 ml/min to 4 ml/min.

Then the fracture conductivity of the proppant can be calculated using Equation (4).

$$KW_f = 1644.84 \frac{\mu QL}{W \Delta P} \quad (4)$$

Where K is fracture permeability (mD), $W_f$ is the fracture width (cm), $KW_f$ is the proppant pack conductivity (mD.ft), μ is the test liquid viscosity at test temperature (centipoise), Q is the flow rate (ml /min), L is the length between pressure ports (cm), W is the cell width (cm), and ΔP is the pressure drop across the sample (bar).

Fig. 2: The Hach 2100N turbidity meter.

Fig. 3: The fracture conductivity cell.

Fig. 4: The Oligocene sandstone core sample platens from Cairo - Elsokhna desert road.

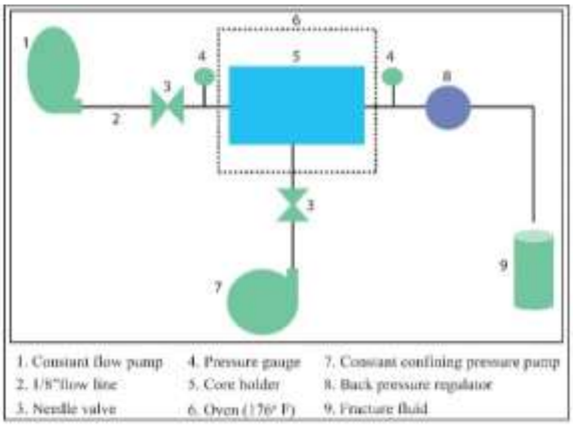


Fig. 5: A schematic illustration for the laboratory setup.

Fig. 6: The Oligocene sandstone platens with proppant before conducting fracture conductivity test.

**2. Results and discussion**

*2.1. The physical properties*

The size of the proppant grains has a major impact on the proppant conductivity with larger grain size typically leads to a greater proppant pack permeability under a lower closure pressure. As the closure pressure increases, the effect of grain size is decreased due to the associated crushing of larger grain sizes. The larger particle sizes are also preferable for being transported without being bridging out during the fracturing treatment. Sieve analyses with a series of standard wire mesh stacked sieves showed that more than 90% of the new polymer Nano-composite proppant particles are in the range of 450 microns in the studied sample. Figure 7 illustrates rod-shaped proppant particles of the new Nano-composite proppant particles under the optical microscope. The rod-shaped proppant provides a higher conductivity compared to the conventional spherical types because of its higher porosity in its packing [39]. The development and evolution of the rod-shaped proppant particles considers many drawbacks of the present technologies applied in the fracturing operation. The rod-shaped proppant technology is not chemically limited as the resin coated proppant type. Indeed, the perfect flowback control of the rod-shaped particles is a main advantage of changing the conventional particle shape as particle shape regulates packing behavior and interactions with grains. The proppant particles configuration decreases the ability of particles to move, and inconsequence reduces the pressure on the proppant pack as compression increases [40].

Fig. 7: Proppant particles shape under the optical microscope.

Bulk density of a proppant depends on particle density and arrangement of particles inside the test cylinder. The average particle density of the studied samples is presented in Table 1 and by substituting in Equation (1) the calculated bulk density is 0.4444 g/ml. Since proppants are usually purchased by mass, the advantage of a particular proppant depends on its volume. For a typical fracturing operation, the proppant density will significantly affect the width of the propped fracture. Denser proppant typical maintains a narrow fracture width, Zoveidavianpoor and Gharibi [41]. Proppant density is inversely propositional to the created fracture conductivity and directly proportional to proppant transport. Consequently, a lower density proppant density has several advantages including easy transportation by low viscosity fracturing fluids (e.g. slickwater), improved fracture width, and decreased demands for polymers in the fracturing fluid. Thus, such characteristics associating low density proppant would improve induced fracture conductivity [42]. Results showed that the bulk density of the new proppant is lower than conventional proppant types such as ceramic (1.53) and RCP (1.46). Lower bulk density of a proppant indicates that fewer grams of proppant particles per fracture volume (cm3) are needed to keep the fracture open [43]. As results are compared to RCP (1.46 g/cm3), ULW-1.25 (0.85 g/cm3) and CMRCP (0.68 g/cm3), the new proppant weight required to fill the same volume of the fracture would be respectively 69%, 47% and 34% less.

Table 1: The results of the bulk density test.

| Volume (ml) | Weight (gm) |
|---|---|
| 100 | 44.16 |
| 100 | 44.76 |
| 100 | 44.4 |
| average | 44.44 |

Specific gravity is considered an essential factor in evaluating the quality of a proppant in fracturing operation. Materials with high strength and lower density are ideal for proppant markets. Materials with low apparent specific gravity will reduce the treatment cost of pumping price due to their high buoyancy in low viscous fracturing liquids (slickwater). In general, the optimal specific gravity is typically less than 2 to consider the proppant as ULWP. The calculated proppant density from laboratory measurement is 0.8 g/cm3. Using the average density of the reference liquid (kerosene) and substituting in Equation (2), the specific gravity of the new proppant is 1.006. This indicates lighter the proppant particles with a greater volume that requires much less proppant mass for a successful hydraulic fracturing treatment. The new proppant has shown lower specific gravity (1.006) compared to conventional types such as frac sand (2.66), RCP (2.80), and ceramic (3.1). Similarly, it also reports lower specific gravity compared to modern types such as CMRCP (1.42) and ULW-1.25 (1.25). Such a comparison is probably enough for considering the new proppant as an ideal ULWP.

*2.2. Mechanical properties*

The Turbidity test reported 28 NTU (Figure 8 Left) for the studied sample (Figure 8 Right). The measured turbidity is attributed to the small fines that occur on the proppant surface, which may clog the pores across the proppant pack and thus decrease the induced fracture conductivity. Compared to other types, the resulting proppant turbidity test is lower than available proppant types including frac sand (100 NTU), ceramic (80 NTU) and CMRCP (38 NTU). This indicates better performance of the new Nano-composite proppant compared to the other proppant types.

The Oil and Gas Industrial Standard (SY/T5108-2006) recommended that the acid solubility of a proppant should be less than 5% by weight. The final weight of the proppant sample after performing the acid solubility test was 4.9 g and by substituting in Equation (3), the acid solubility of the new proppant is calculated as 2%. This result is almost equivalent to that of CMRCP (1.8%) and better than the conventional types such as ceramic (5.89%) and silica sand (4.59%). Lower acid solubility indicates higher corrosion resistance and inconsequence higher stability in the acidic environment with a greater ability to maintain a long-term conductivity [44].

Fig. 8: The result of the turbidity test (Left), and the tested sample (Right).

*2.3. The fracture conductivity test*

The importance of fracture conductivity and its effect on the well productivity is quite understood in the oil and gas industry [45]. The results of five different injection rates (2, 2.5, 3, 3.5 & 4 ml/min) at several closure pressure (1000, 2000, 3000 & 4000 psi) and four different pressure drops for each injection rate-closure pressure combination were recorded, Table 2.

The measured liquid viscosity at the test temperature μ was approximately 0.399 cp. and the geometric dimensions of the Oligocene sandstone core sample were (length: 6.77 cm) and (width: 3.75 cm), as measured by a Vernier caliper. By substituting these parameters in Equation (4), the fracture conductivity is directly calculated using Equation (5) and experimental results.

$KW_f = 1184.8\frac{Q}{\Delta P}$ (5)

Where $KW_f$ is the proppant pack conductivity (mD. ft), Q is the flow rate (ml /min), and ΔP is the pressure drop (bar).

Table 2: The results of fracture conductivity test.

| Closure pressure psi | Flow rate ml/min | ΔP bar | ΔP psi | Fracture conductivity mD.ft |
|---|---|---|---|---|
| 1000 | 2.0 | 0.18 | 2.60 | 13164.7 |
| 2000 | 2.0 | 0.30 | 4.40 | 7898.8 |
| 3000 | 2.0 | 0.45 | 6.50 | 5265.9 |
| 4000 | 2.0 | 0.75 | 10.90 | 3159.5 |
| 1000 | 2.5 | 0.23 | 3.30 | 12878.5 |
| 2000 | 2.5 | 0.45 | 6.50 | 6582. 4 |
| 3000 | 2.5 | 0.70 | 10.20 | 4231.5 |
| 4000 | 2.5 | 1.05 | 15.20 | 2821.0 |
| 1000 | 3.0 | 0.30 | 4.40 | 11848.3 |
| 2000 | 3.0 | 0.65 | 9.40 | 5468.4 |
| 3000 | 3.0 | 1.00 | 14.50 | 3554.5 |
| 4000 | 3.0 | 1.40 | 20.30 | 2538.9 |
| 1000 | 3.5 | 0.40 | 5.80 | 10367.2 |
| 2000 | 3.5 | 0.90 | 13.10 | 4607.7 |
| 3000 | 3.5 | 1.40 | 20.3 | 2962.1 |
| 4000 | 3.5 | 2.00 | 29.0 | 2073.5 |
| 1000 | 4.0 | 0.70 | 10.2 | 6770.4 |
| 2000 | 4.0 | 1.10 | 16.0 | 4308.5 |
| 3000 | 4.0 | 2.10 | 30.5 | 2256.8 |
| 4000 | 4.0 | 2.50 | 36.3 | 1895.7 |

The experimental results presented in Table 2 indicate a direct proportion relationship between the closure pressure and the pressure drop at the same flow rate. The rate of increase in the pressure drop from 1000 to 2000 psi of closure pressure is 67 % at constant flow rate of 2 ml/min. As the closure pressure increases from 2000 to 3000 psi at the same flow rate (2 ml/min) resulted in increasing in the pressure drop by 50 % (Table 2). A similar observation is reported in other flow rates with different closure pressures. The results also show a direct proportional relationship between flow rate and the pressure drop at the same closure pressure. The rate of increase in the pressure drops while increasing the flow rate from 2 to 2.5 ml/min is about 28 % under the same closure pressure of 1000 psi. Increasing the flow rate from 2.5 to 3 ml/min under the same closure pressure (1000 psi) caused an increase in the pressure drop by 30 %. From these results, one can conclude that the increased flow rate or velocity of fluids injection can cause great amounts of energy to be lost as the fracturing fluid moves through the proppant pack. This is due to the inertia effect caused by changes in fluid flow directions. The phenomenon of inertial effect can be described simply by simulating a racing car driver. The driver can travel around the path on a very simple track if he chooses to travel at a very slow speed. Minimum energy is lost in changing the car direction at turns. However, if the driver decides to drive as quickly as possible, he will slow down in turns and accelerate on the straight ways. Every time he turns, the energy in the break is lost as well as the forward momentum is lost. The fluids moving through the flow paths act in a similar fashion to that described by the racing car. Using Equation (5) for the recorded data, the fracture conductivity was calculated for each closure pressure as shown in Table 2, and the average conductivity value of the five flow rates was calculated for each closure pressure and the results are shown in Table 3. Figures 9 and 10 show the images of the core sample and proppant, respectively, after the experiment has finished and releasing the applied pressure. Under each of the closure pressure and flow rate different fracture conductivity is reported. Fracture conductivity decreases from 13164 to 3159 mD.ft as the applied closure stress increases from 1000 to 4000 psi at a constant flow rate of 2 ml/min. As flow rate increases from 2 to 2.5 ml/min, fracture conductivity significantly falls from 12878 to 2821 mD.ft for the same closure pressure. Under a constant closure pressure of 1000 psi, fracture conductivity decreases from 13164 to 6770 mD.ft (almost 50 % declining) as the flow rate increases from 2 to 4 ml/min. A similar trend takes place under 4000 psi applied closure stress, as fracture conductivity decreases from 3159 to 1895 mD.ft as injection flow rate increases from 2 to 4 ml/min. These changes show inverse proportional relationships of fracture conductivity to injection flow rate, closure pressure and pressure drop. As the applied closure stress increases from 1000 to 2000 psi, the fracture conductivity decreases by 52% (Table 3). A similar trend is taking place when increasing closure stress from 2000 to 3000 psi with 63% decline in fracture conductivity. Finally, as closure pressure increased from 3000 to 4000 psi, fracture conductivity decreased by 68% till reached 2497 mD.ft (Table 3).

Table 3: The average conductivity of five flow rates for each closure pressure.

| Closure pressure psi | Average fracture conductivity mD.ft |
|---|---|
| 1000 | 11005.8 |
| 2000 | 5773.2 |
| 3000 | 3654.2 |
| 4000 | 2497.7 |

Fig. 9: The core sample after conducting the lab experiment.

Fig. 10: The proppant after the lab experiment.

The propped fracture conductivity with the new proppant showed better performance compared to other proppant types. The bulk density of the new proppant is less than the conventional proppant types and less than the other two comparative types. This means lower weight of the new proppant will provide more volume compared to other types. The rod-shaped particles provide a higher conductivity because of the associating higher porosity of proppant packing. Figure 10 show the high crush resistance of the new proppant and therefore generating fines that decrease the proppant pack conductivity will be diminished. Accordingly, the new proppant provides a strong structure that withstand against high closure pressure compared to other proppant types.

The results of this study were compared to the results of two published studies. The first study was conducted in 2006 by Rickards et al. that showed the possibility of using walnut-hull (ULW-1.25) particles as a propping agent. The second study was carried out in 2018 by Mansoor et al. on a new proppant called CMRCP (Chemically Modified and Reinforced Composite Proppant) which was derived from renewable resources. The comparison of the three studies is shown in Figure 11. In all three cases, the fracture conductivities decrease as the applied closure pressure increases. Unlike the ULW-1.25 proppant with a fracture conductivity of 6240 mD.ft, the new polymer-Nano-composite and CMRCP proppants have very good conductivities (11000 and 12250 mD.ft, respectively) at the earlier pressure step (1000 psi). These results indicate that the CMRCP and the new polymer-Nanocomposite proppant have similar, somehow high performance at low closure pressures. Alternatively, at effective closure pressure from 1000 to 3000 psi the new Nano-composite proppant exhibited much higher fracture conductivity than ULW-1.25 but like CMRCP. The behavior of the three proppant types under stress exhibited similar trends (Figure 11). As pressure increases from 1000 to 2000 psi, fracture conductivity decreases rapidly by 60%, 52% and 53% in CMRCP, the new polymer-Nanocomposite proppant and ULW-1.25, respectively. Other notable decreasing trends in fracture conductivity are observed under closure pressure from 2000 to 3000 psi with a rate of decrease of 68%, 63% and 65% for CMRCP, the new polymer-Nanocomposite proppant and ULW-1.25, respectively. As the closure pressure increases from 3000 to 4000 psi, the decline in fracture conductivity is still similar to the pervious trends. Thus, the new polymer Nano-composite proppant can provide similar or better fracture conductivity than the other proppant products.

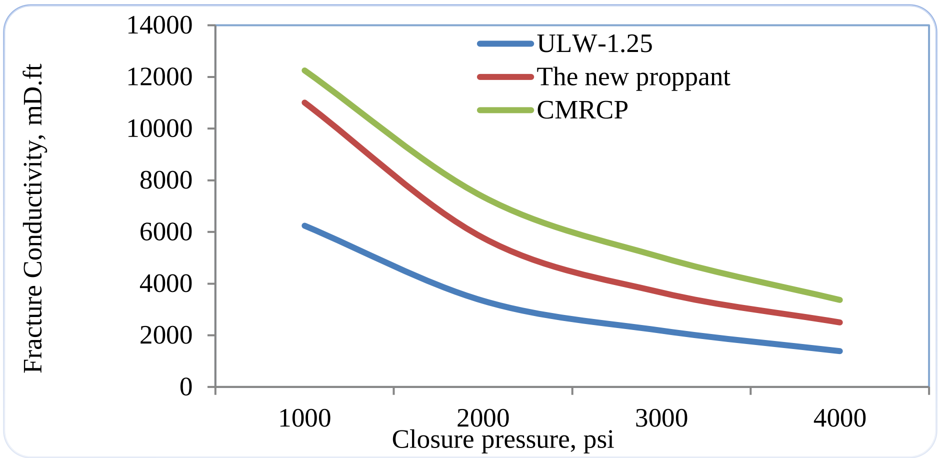


Fig. 11: A comparison of fracture conductivity tests for CMRCP, the present study new polymer-Nanocomposite, and ULW-1.25 proppants.

Many mechanisms such as fines migration, crushing and proppant diagenesis, and the embedment of proppant may participate in fracture conductivity reduction. Each mechanism plays a role in decreasing the conductivity of the induced fracture and usually varies according to the mechanical properties of the formation, formation type, the type of fracturing fluids, the closure stress, the mineral content of formation, and the proppant type [46]. The new polymer Nano-composite proppant showed enhanced conductivity results as compared to CMRCP and ULW-1.25 (Figure 11). Such enhancement is attributed to improvement of crush resistance related to the enhanced physical and mechanical properties of the agro-waste substrate. In addition, polymer and nanoparticles played an essential role for the enhancement of conductivity. Since the new proppant has a specific gravity of 1.006 and a bulk density of 0.44 g/cm3, the manufacturing method of the new Nano-composite proppant has been contributed to enhancement of fracture conductivity at different closure pressures. Another comparison to fracture conductivity of the new polymer Nano-composite proppant with many widely used proppant types [46] is illustrated in Table 4. The fracture conductivity of the new polymer Nano-composite proppant under closure pressure of 4000 psi is more than walnut shell, palm kernel shell, and silica sand, whereas it is close to that of coconut shell (Table 4). The new polymer Nano-composite proppant also has the ability of providing higher conductivity compared to Brady sand and ULW-1.25. However, its performance is not as high as CMRCP and ceramic proppants (Table 4). The high fracture conductivity of the CMRCP could be related to its lower acid solubility (1.8%), because further percentage of the smaller fines and impurities reduces proppant pack permeability.

On comparing the present study results to the results reported by Zoveidavianpoor and Gharibi [41] for measurements conducted on Walnut shell, Palm Kernel shell and Coconut shell for potential use as propping agent in hydraulic fracturing treatment, the new polymer Nano-composite proppant has shown much higher conductivity values at lower closure pressures (up to 2000 psi) but as closure pressure increases the fracture conductivity decreases compared to Palm kernel and Coconut shells, Figure 12. The ability of the new polymer-Nano-composite proppant to provide better fracture conductivity can be related to the high crush resistance resulting from adding the polymer and nanoparticles to its substrate (rice husk) that generates less fines.

Table 4: The comparison of fracture conductivity of the new polymer Nano-composite proppant with other proppants [47].

| Proppant type | Fracture conductivity (mD.ft) | Closure pressure (psi) |
|---|---|---|
| The new Nano-composite proppant | 2497 | 4000 |
| CMRCP | 3368 | 4000 |
| ULW-1.25 | 1386 | 4000 |
| Sand | 388 | 5000 |
| Brady Sand | 4000-35 | 2000–10,000 |
| RCS | 386 | 10,350 |
| Ceramic (ELWC) | 619 | 11,960 |
| Ceramic (ELWC-Upgrade) | 1170 | 10,350 |
| SMA (surface modifying agent) | 95 | 6000 |
| Intermediate strength proppant (ISP) | 12,000 | 4500 |
| Palm kernel shell | 2269 | 4000 |
| Walnut shell | 1885 | 4000 |
| Coconut shell | 2558 | 4000 |
| Coated porous ceramic (ULW-1.75) | 4523-487 | 2000–8000 |
| High Strength Proppant (HSP) | 35,000–4000 | 2000–10,000 |
| Intermediate Strength Proppant (ISP) | 22,000–3000 | 2000–10,000 |

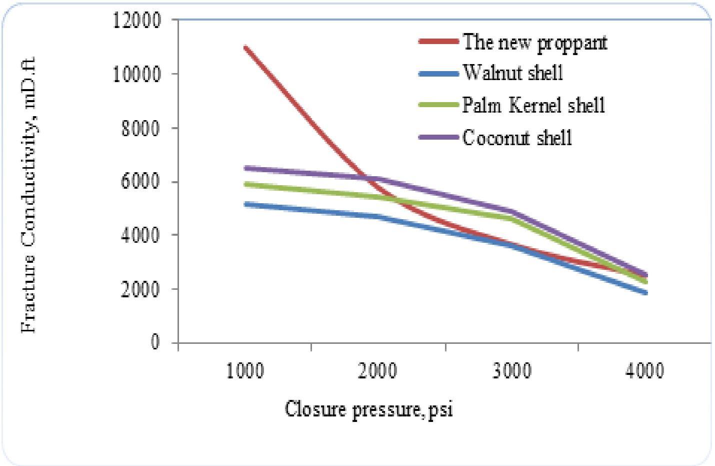


Fig. 12: Fracture conductivity behavior for the new polymer-Nanocomposite, Walnut shell, Palm Kernel shell, and Coconut shell proppants as closure pressure increases.

The propping agents with low crush resistance will be crushed if subjected to high closure stresses, and the crushed proppant particles generate fines that decrease fracture conductivity of the induced fracture [48]. The improved fracture conductivity achieved by the new Nano-composite proppant can be attributed to the lower acid solubility (2%) and low turbidity (28 NTU), as additional fines and impurities will decrease proppant pack conductivity. Finally, the shape of the proppant (rod-shaped) improves the proppant pack permeability.

### 3. Conclusion

The present study discusses the results of experimental characterization of a new polymer Nano-composite propping agent according to the procedures of testing and evaluating the conductivity of a new proppant agent (ISO 13503-5, API RP 56). The use of deformable substrates as proppant with scattered distribution contributes to enhancement of the fracture conductivity in hydraulic fracturing treatment. As high closure stresses apply on the new proppant, rather than crushing, it deforms reasonably. The new polymer Nano-composite proppant reported an apparent specific gravity of 1.006 and a bulk density of 0.44 g/cm3. The present study revealed excellent physical and mechanical properties of the new polymer Nano-composite proppant. Low specific gravity, rod-shaped form, low acid solubility percentage, low bulk density, low turbidity value, high crush resistance and relatively better conductivity have been qualified the new proppant to be considered as a new ULWP. It has perfect properties, superior performance at high closure pressures and enhanced temperature tolerance, as compared with conventional proppant types, ULW-1.25, and other ULW proppant types.

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