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Research article

Effect of using Austrian pine cones powder as an additive on oil well cement properties

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A R T I C L E I N F O

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A B S T R A C T

There have been many investigations to improve both the physical and mechanical properties of oil well cement using a wide range of materials. Most of these additives are expensive and practically ineffective. In this article, a comprehensive evaluation was conducted for using Austrian pinecones powder (APCP) as an inexpensive supplementary cementing material (SCM) for well cement. Firstly, Portland cement class G was characterized based on X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD). In this paper, the properties of the cement systems include rheological parameters, density, slurry stability (free water test, and sedimentation test), water absorption, porosity, permeability, the volume of fluid loss, pH value, thermogravimetric analysis, and the mechanical characteristics (in terms of compressive strength, tensile strength, flexural strength, and shear strength bond) were investigated in details. The cement sample containing the APCP was also examined using scanning electron microscopy (SEM). According to the experimental results, adding APCP led to increasing in rheological parameters. Also, led to decreasing in fluid loss, free water, sedimentation effect, and density which positively affects the preservation of the original properties of cement slurry. The results also showed a decrease in the permeability of cement samples and an increase in the porosity and the ability to absorb water. The addition of APCP did not significantly affect the pH values. The addition of APCP also deteriorated the mechanical properties of the cement samples. The addition of the APCP has contributed to an increase in total weight loss at high temperatures. So, the APCP can be considered as a new filler for well cement due to its ability to fill the pores in the cement matrix and at the same time improve some properties of the well cement such as density, free water, sedimentation, and fluid loss.

1. Introduction

In order to move the wheel of industry in the world, oil and gas are needed. The oil, and gas industry is one of the most important industrial sectors in the world. This industry involves the implementation of many operations to produce oil and gas. Well drilling is considered one of the most important operations. During drilling operations, it is necessary to isolate the drilled formations by pumping one or more cement slurries into the annulus between the casing string and formations after the drilling of each section is

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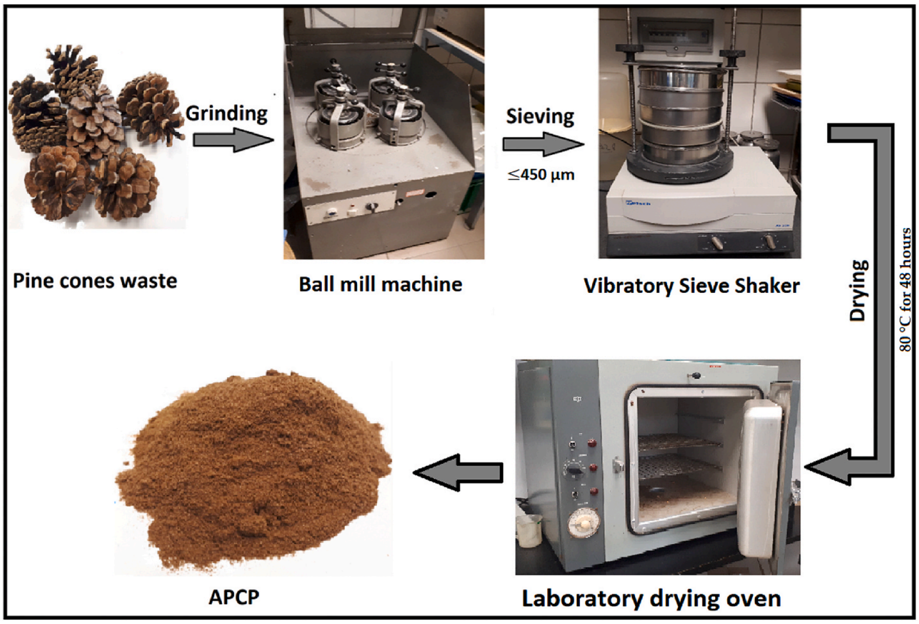

Fig. 1. Manufacturing process to produce APCP.

completed [1]. Therefore, the properties of this cement sheath are the main key to the success of the drilling and production operations [2]. Several problems may occur in the oil well as a result of cement sheath failures such as the corrosion of the casing and migration of the gas to the surface resulting in a loss of well integrity [3]. Therefore, well integrity is one of the main challenges facing the oil and gas industry. Borehole integrity failure has serious environmental consequences, such as contamination of freshwater zone near the surface, the release of toxic hydrocarbon gases into the atmosphere, and leakage of hydrocarbon liquids up toward the surface. In addition to these problems, terrible damage can occur to the casing string and lead to an increase in the repair activities and the cost of maintenance [4]. Thus, the design of the cement mixture plays a major role in maintaining the integrity of the well. Several solid and liquid chemical additives are usually used to control the different properties of the cement slurry. These additives can control the rheology, density, thickening time, compressive strength, fluid loss volume, porosity, permeability, and free water separation [5]. Portland cement is the main element of cementing sheath, but it cannot withstand the high stress of the downhole when employed directly in cementing operation after being mixed with water because it has low tensile strength and high cement shrinkage after dehydration [6]. As a result, it is frequently required to use additives to control cement slurry properties. Accelerators, retarders, extenders, and weighing agents are the most typical additives to well cement [7].

In the development of well cement systems, mechanical properties are considered the most important factor [8–10]. Improving the compressive strength and cement-casing bonding strength are the main factors to performing a successful cementing job and maintaining the integrity of cement and enhancing the ability to withstand imposed stresses and preventing cement sheath failure.

Recently, researchers focused on conducting many experimental studies including chemical and physical analyses on a wide range of industrial and agro-waste materials have revealed properties that give them an advantage for use as potential cement additives. Those materials included: eco-friendly waste materials such as olive waste as in the study conducted by Ref. [11] which showed Enhancement in the durability of Saudi Class G oil-well cement sheath in CO2 rich environments after using olive waste. In addition to a significant decrease in the permeability of cement samples by 14.3% less than the reference cement sample after adding 0.1% by weight of cement (BWOC) of olive waste to the cement slurry, eggshell waste as in the study conducted by Ref. [8] which showed improvement in mechanical properties of well cement and supported the hydration process, and rice husk ash as in the study conducted by Ref. [12] which showed a significant improvement in some properties after adding rice husk ash as pozzolan to cement at different concentrations. But using non-renewable resources in oil well cement makes the process unsustainable. Based on the United Nation's sustainable development goal number 12 ask for sustainable practices in the industry [13]. Lime decarbonisation, which occurs during manufacturing clinker (the primary in-process ingredient in cement production) led to a 5% increase in global Carbon dioxide emissions [14]. The oil and gas industry has tried to support sustainability over the years by encouraging the utilization of green materials in its different operations. Thus, using eco-friendly materials has a positive effect not only because of their low cost but also for their low negative impact on human health and the environment if used as an alternative to industrial chemical additives to control the properties of cement slurry [15].

Waste pine cones are one of the most common agroforestry wastes in nature. The part of the pine tree that contains the seeds is called a pine cone [16]. The main chemical compositions of pine cones are cellulose, hemicellulose, and lignin [17]. These cones have no practical application in industry and are only used for decoration and the manufacture of some toys. Some researchers have tried to employ these natural fibers in some applied fields [18,19]. In the oil and gas industry, the possibility of using pine cone powder as an

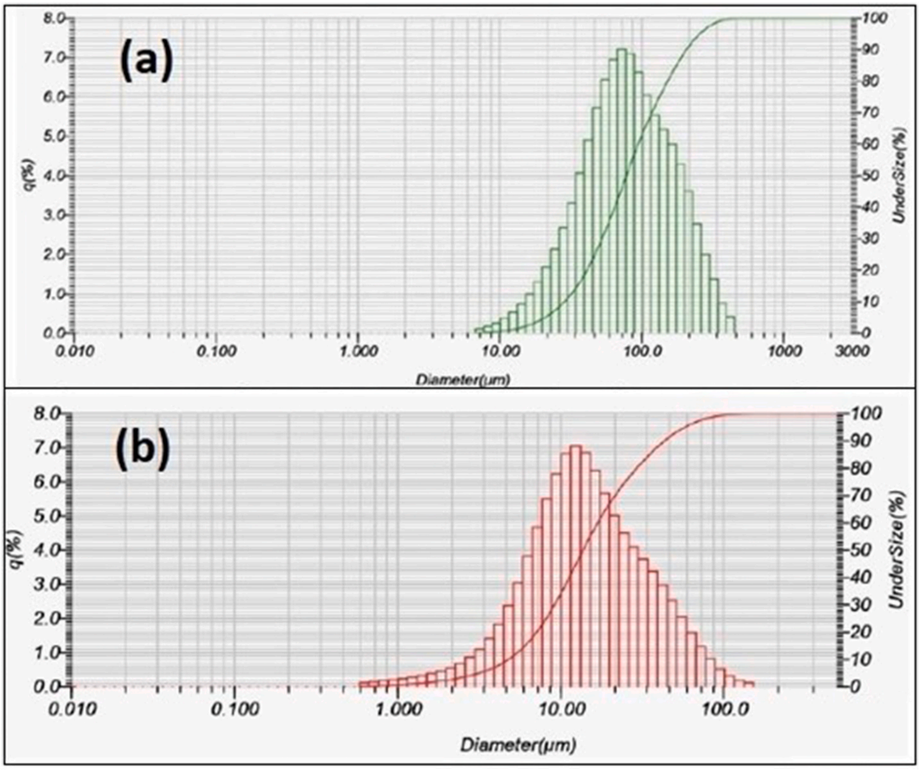

Fig. 2. The particle size distribution of (a) the APCP and (b) the neat cement powder.

Table 1
The physical properties of APCP and cement were used for the purpose of this study.

Powder type	Color	Median particle (μm)	Mean particle size (μm)	Mode particle size (μm)	Geo. Mean particle size (μm)
APCP	Brown	77.98	100.62	72.35	77.02
Cement	Gray	13.72	19.86	12.38	13.87

additive to water-based drilling fluids has been investigated [20]. Tonnes of pine cones are produced every year around the world and are still environmentally neglected [16]. To the best of our knowledge, this research is the first attempt to employ waste pine cones as a supplemental cementing material (SCM) in the petroleum industry. The extraction of resin from pine trees is what encouraged the authors to perform this study. The resin and its emulsions are used as an additive to the cement matrix of concrete and well cement to improve the mechanical properties [21,22]. Consequently, our work is crucial in emphasizing the use of these natural fibers in practical fields.

The primary objective of this study is to evaluate the applicability of using different concentrations of APCP as an additive to class G oil well cement by performing a series of measurements, afterwards suggesting some recommendations for potential applications of this material in oil well cementing.

2. Materials and research methodology

2.1. Preparation of the APCP

Waste Austrian pine cones were obtained from the Miskolc city of the Borsod-Abauj-Zemplen county in Hungary. These cones were cleaned with clean water and should be dried under the sun for one week before starting the APCP manufacturing process. The cones open after drying and then the seeds can be removed easily. The cones were milled using a ball mill machine. Thereafter, the cones powder was sieved through a mesh with a size of 450 μm or less by using Retsch Vibratory Sieve Shaker AS 200. It is the smallest particle size that can be obtained because of the high temperatures recorded during cones grinding by the ball mill machine. Finally, the powdered cones were dried in an oven at 80 °C (which have been selected experimentally) for 48 h to reduce the humidity. The reason for this is that the humidity content may have a negative impact on research results, especially in slurry stability and fluid loss volume tests. The procedures of manufacturing process to produce APCP are depicted in Fig. 1. The particle size distribution of

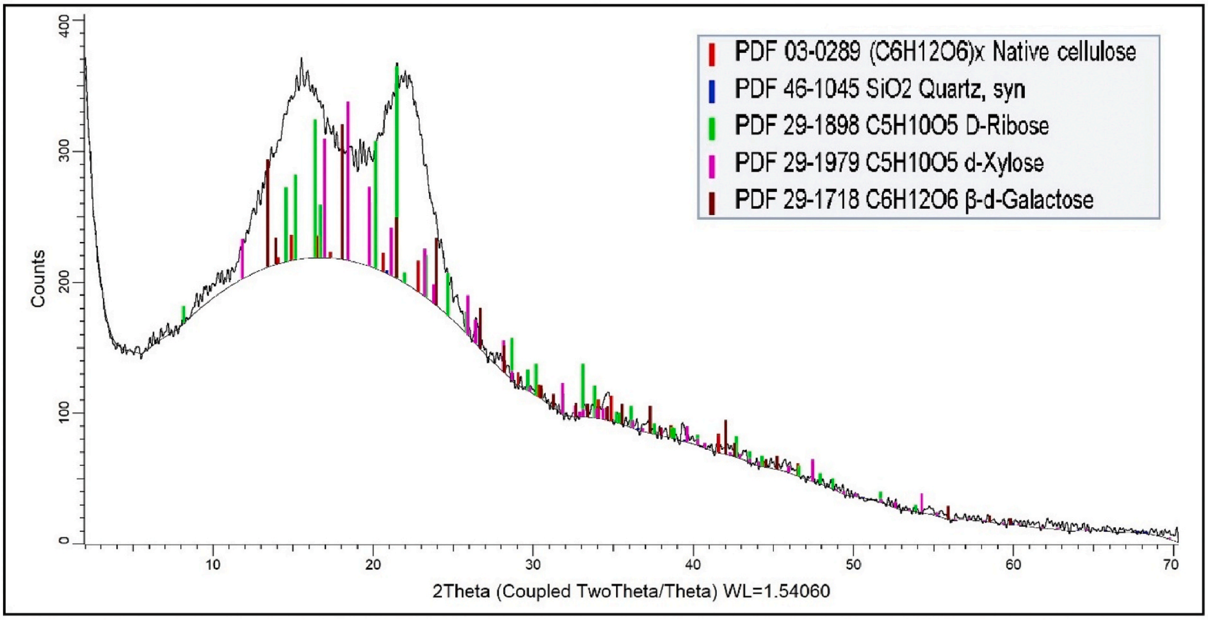

Fig. 3. XRD analysis results for APCP.

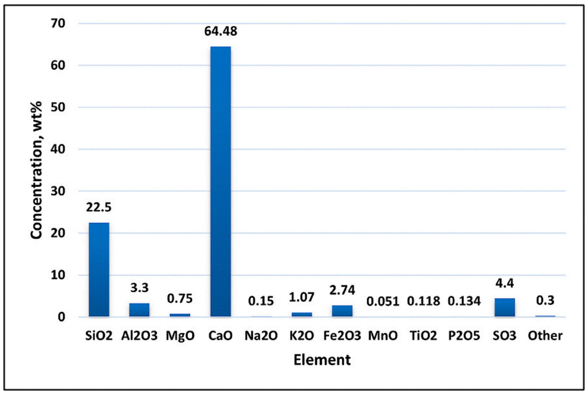

Fig. 4. Elemental composition of class G well cement by using XRF analysis.

pinecone powder and neat cement powder were analyzed using the HORIBA LA-950V2 laser diffraction particle size analyzer as shown in Fig. 2. It is important to mention that Fig. 2a represent the particle size distribution of APCP and Fig. 2b represent the particle size distribution of neat cement powder. The x-axis depicts pine cone particle diameter, whereas the y-axis depicts cumulative undersize particles (%). The physical properties of APCP and neat cement powder are presented in Table 1. Similar to the method used by Ref. [23] to measure bulk density, the results showed that the bulk density of APCP was 0.4788 (g/cm^3). The X-ray diffraction analysis (XRD) of the APCP were given in Fig. 3. According to the results of (XRD) analysis, APCP mainly consists of native cellulose, D-Ribose, D-Xylose, and ß-D-Galactose. It can be noticed that there is a small amount of quartz as a result of contamination of the cones with the sand stuck to them, despite the process of cleaning and rinsing it with clean water several times.

2.2. *Preparation of the cement slurry samples*

A standard API recipe of Class G well cement based on (API 10A/ISO 10426-1, 2009) has been used in this study as a reference sample. Class G cement is the most common type of Portland cement for cementing wells. The water-to-cement ratio used to prepare the cement slurries was (W/C = 0.44) based on the recommendation of API standards. Distilled water was used to prepare the cement slurry since tap water contains chloride sulfate and other impurities that can attack cement, it can change the hydration of the cement and affect its strength or other factors. There was no requirement for a dispersant, anti-foam, or any type of cement additives for this study other than Portland cement Class G and the APCP. This was due to the low concentrations of APCP used to improve the different well cement properties. Fig. 4 summarizes the chemical composition of class G cement determined by X-ray fluorescence (XRF). X-ray

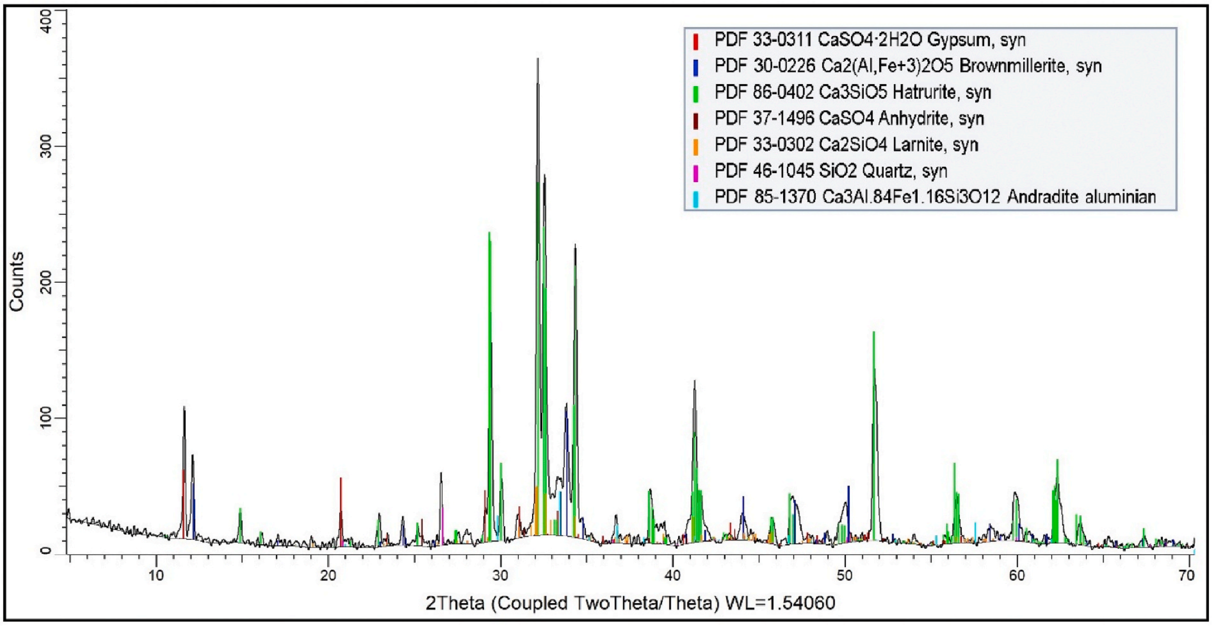

Fig. 5. XRD analysis results for the neat Class G cement.

Table 2
Cement slurries composites were used in this study.

Cement slurry	Concentration of APCP (%BWOC)	Weight of APCP (g)	Weight of cement class G (g)	Distilled water (g)
Slurry I (neat cement)	0	0.00	792	349
Slurry II	1	7.92	792	349
Slurry III	2	15.84	792	349
Slurry IV	3	23.76	792	349
Slurry V	4	31.68	792	349
Slurry VI	5	39.60	792	349

powder diffraction (XRD) had been used to determine the mineralogy of the neat cement powder using a Bruker D8 Discover XRD SAXS XRR with long-fine focus tube radiation operating at 50 kV voltage and 50 mA current as shown in Fig. 5. Cement slurry preparation and a series of experiments were performed according to API standard 10 A specifications [24] and API RP 10-B2 [25]. All cement slurry proportions used in this study are listed in Table 2. To prepare the cement slurry, manual mixing for 5 min was used to mix the dry components including cement powder, and the designed concentrations of APCP in order to obtain homogeneous distributions of APCP. The resulting dry mixture was gradually poured into the bowl of OFITE WARING commercial blender containing distilled water during mixing at low speed 4000 rpm for 15 s. The blender mixing speed as a next step was increased to 12000 rpm for 35 s until a homogeneous cement slurry was achieved. The mixing process was done at atmospheric pressure and room temperature (24 °C). All experiments were performed on the studied concentrations of APCP and then compared with the neat cement sample.

2.3. Determination of rheological properties

These measurements are essential for this evaluation study due to the addition of APCP can significantly affect the workability of the cement slurry. The rheological measurements of cement slurry samples were carried out using a Fann-35 viscometer according to API RP 10-B2 standard. Recording the dial reading at six readings from a lower speed to a higher speed (3 rpm, 6 rpm, 100 rpm, 200 rpm, 300 rpm, and 600 rpm). The dial reading was used to calculate the shear stress against shear rate and viscosity by using **Equations (1)–(3)**. It's worth noting that each test was conducted twice for all cement slurries to verify that the results were consistent.

$$\eta = \frac{\tau}{\gamma} \tag{1}$$

$$\tau = 1.065 \times \Theta \tag{2}$$

$$\gamma = 1.7023 \times N \tag{3}$$

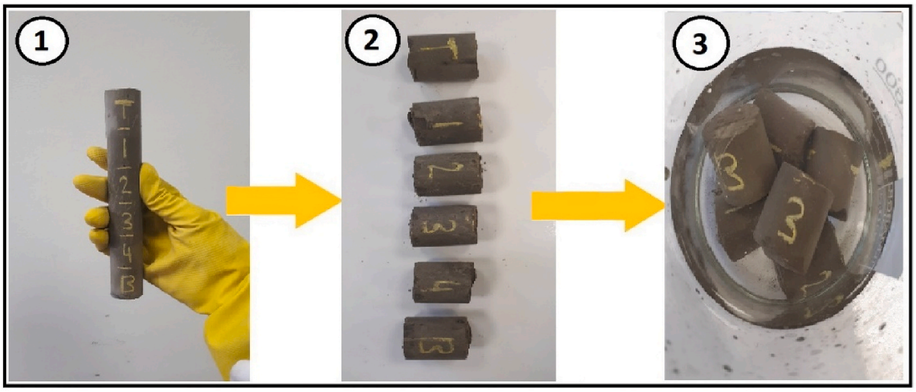

Fig. 6. Sedimentation test (1) the cement sample before cutting (2) the segments of the cement sample after cutting (3) segments immersed in a water bath.

where η is the viscosity (Poise), τ is the shear stress (dynes/cm^2), ϴ is the Fann viscometer reading, γ is the shear rate (1/s), and N is the rate of revolution of the outer cylinder (rpm).

2.4. Determination of slurry stability

The free water test and the sedimentation test, both outlined in API Recommended Practice 10B-2 [25], are used to determine cement slurry stability. Fluids that have higher free fluids or more particles sedimentation are considered to be less stable.

It is a standard laboratory test to measure the volume of separated water on the top of the cement slurry at a static state for a certain period and carried out based on API RP10B-2. The maximum free-water content according to API standard 10A specifications is 5.9% [26]. The free water can be calculated as a percentage by using the following **Equation (4)** [27]:

$$\varphi = \frac{VF}{Vs} \times 100 \tag{4}$$

Where V_F is the volume of free water collected (mL), and Vs is the initial volume of the cement slurry (mL).

The sedimentation test was carried out according to Ref. [25] standards. The segments were numbered (Top, 1,2,3,4, and bottom) after cutting the cement sample column into six equal segments as shown in Fig. 6a, b, and c. These segments separately were weighed in the air and then weighed in the water. Then, the following formula **(5)** can be used to calculate the density of each segment:

$$\rho = [(Wa) / (Wa - Ww)] \times \rho w \tag{5}$$

where ρ is the density of segment in (g/cm^3), Wa is the weight of segment in air in (g), Ww is the weight of segment in water in (g), ρw is the density of the water (g/cm^3).

Finally, the density difference ($\Delta\rho$) between the top segment (ρtop) and bottom segment ($\rho bottom$) can be calculated by the following **Equation (6)**:

$$\Delta\rho = \rho bottom - \rho top \tag{6}$$

2.5. Determination of density

The Model 140 Fann Mud Balance was used for measuring the density of the cement slurry. The mud balance cup is filled with cement slurry immediately after preparation. The cup was closed with a lid and afterwards cleaned from outside due to the cement coming out of the lid hole. Thereafter, the cup was then balanced by placing it on a fulcrum and adjusting a sliding weight until both sides were balanced. The density of the cement can be read off from the ruler on the device arm and expressed in (lb/gal). It's worth noting that each test was conducted twice for all cement slurries to verify that the results were consistent.

2.6. Determination of fluid loss

This experiment was carried out by using a standard API low-pressure/low-temperature filter press device under static conditions. The cylindrical cell was filled with cement slurry and closed tightly after placing the filter paper. A low pressure of (100 ± 5 psi) from a nitrogen gas bottle was applied to the cell after installing it on the holder of the device. The amount of filtered fluid was collected for 30 min by using a graduated cylinder were placed below the device cell and expressed in (mL). It's worth noting that each test was conducted twice for all cement slurries to verify that the results were consistent.

2.7. Determination of porosity and permeability

Porosity and permeability were measured using cylindrical cement samples after aging them for 28 days in the distilled water bath. Distilled water was used to cure the cement samples since tap water contains chloride sulfate and other impurities that can attack cement, it can change the hydration of the cement and affect its strength or other factors. Nitrogen gas permeability measurements were used to determine permeability, and the Klinkenberg equation was used to calculate the sample's absolute permeability. Using a QUANTACHROME 1200e Helium Pycnometer with a measured volume accuracy of ± 0.02%, the porosity of the samples was determined. The measurement time took an average of 20 min per sample. Then, applying Boyle's ideal gas law at low pressures by using the following **Equation (7)** [28]:

$$P1.V1 = P2.V2 \tag{7}$$

where P1 and V1 are the initial pressure and volume, respectively. P2 and V2 are the pressure and volume of the gas after change, respectively.

2.8. Determination of water absorption, and bulk density

In this test, the cylindrical cement samples (36 mm inner diameter and 64 mm length) after preparing were immersed in a distilled water bath for 28-days curing time at room temperature [29]. The cement samples were removed from the water bath and placed in an oven at a temperature of 80 °C for 24 h. As a next step, the dry samples were weighed in the air after being taken out of the oven. After that, the samples were immersed for 2 h in distilled boiling water, with the water level kept above the submerged samples. Then the cement samples were removed from the water, wiped with soft tissue paper, and weighed in the air on a digital scale, as well as weighed in water. Within 1 min after the cement samples were removed from the water, the measurement process was completed. The average test results on three samples from each concentration shall constitute the test result. Thereafter, the water absorption [%] according to standard test methods for water absorption, and bulk density of burned refractory brick and shapes by boiling water [30] can be calculated by using the following **Equation (8):**

$$Water\ Absorption = [(Ws - Wd) / (Wd)] \times 100 \tag{8}$$

The bulk density [g/cm^3] according to ASTM C20 [30] can be calculated by using the following **Equation (9):**

$$Bulk\ density = [Wd / (Ws - Wsu)] \tag{9}$$

where Wd, Ws, and Wsu are dry weight, saturated weight, and suspended weight of the sample, respectively.

2.9. Determination of pH

The pH of the cement was measured by a digital laboratory pH/mV meter with an accuracy of ±0.01 pH. After aging for 28 days, the inner core of broken samples resulting from the compressive strength test were ground into powder by a ball mill for 15 min. The particles were then sieved to less than 100 μm using a mesh to increase the total surface area of the particles, allowing for more chemical reactions. In clean containers, 100 ± 0.1 g of these powders containing different concentrations of APCP were mixed separately with 200 ± 0.1 g of fresh distilled water and gently stirred for 10 min until the powder is dissolved. The solution was left for 72 h at normal conditions of pressure and room temperature (24 °C) with covering the containers to prevent evaporation. Then, the solution was filtered through filter paper to obtain 50 mL of the filtered solution poured into a clean beaker for pH measurement. Next, the pH electrode calibration should be performed using three standard values of pH buffer solutions which are (pH = 4.00, pH = 6.86, and pH = 9.18) before being used in the test. Thereafter, the pH of the filtered solution was measured to understand the effect of the APCP on the pH value of the cement. By immersing the pH meter in the tested solution and moving continuously the pH meter gently inside it and waiting for 30 s till the reading stabilized before recording the pH value. The test was repeated three times for each solution, and the average value was recorded to verify the accuracy of the results. It is important to rinse the electrode of the pH meter between measurements of different solutions with distilled water and dry it with filter paper.

2.10. Determination of unconfined compressive strength (UCS), tensile strength (TS), and flexural strength

In this type of measurement, cement samples were cured into cylindrical molds with a diameter of 36 mm and a height of 64 mm. For tensile strength test, cement cylindrical samples of 100 mm in diameter and 50 mm in length were used [31–33]. The flexural strength testing was performed on 40 × 40 × 160 mm beams with a 100 mm supported span under three-point loading. The tensile strength and flexural strength tests of oil well cement is not yet standardized at atmospheric or downhole conditions. For this reason, it was carried out following the standards designated for concrete testing in the construction industry [34]. Firstly, the inner faces of the cement molds were cleaned and should be lubricated with a non-reactive releasing agent to make samples removal after curing easier. Thereafter, the cement slurry was poured into the molds with three samples from each concentration for each age, and to avoid any bubbles inside the cement samples should be monitored for a few minutes. All samples were aged for 7, 14, and 28 days in a water bath at room temperature (24 °C) and at atmospheric pressure conditions. Afterwards, the samples were removed from the water bath and the surface of the top and base of the samples were polished and reducing its roughness by using abrasive papers. A vernier caliper was

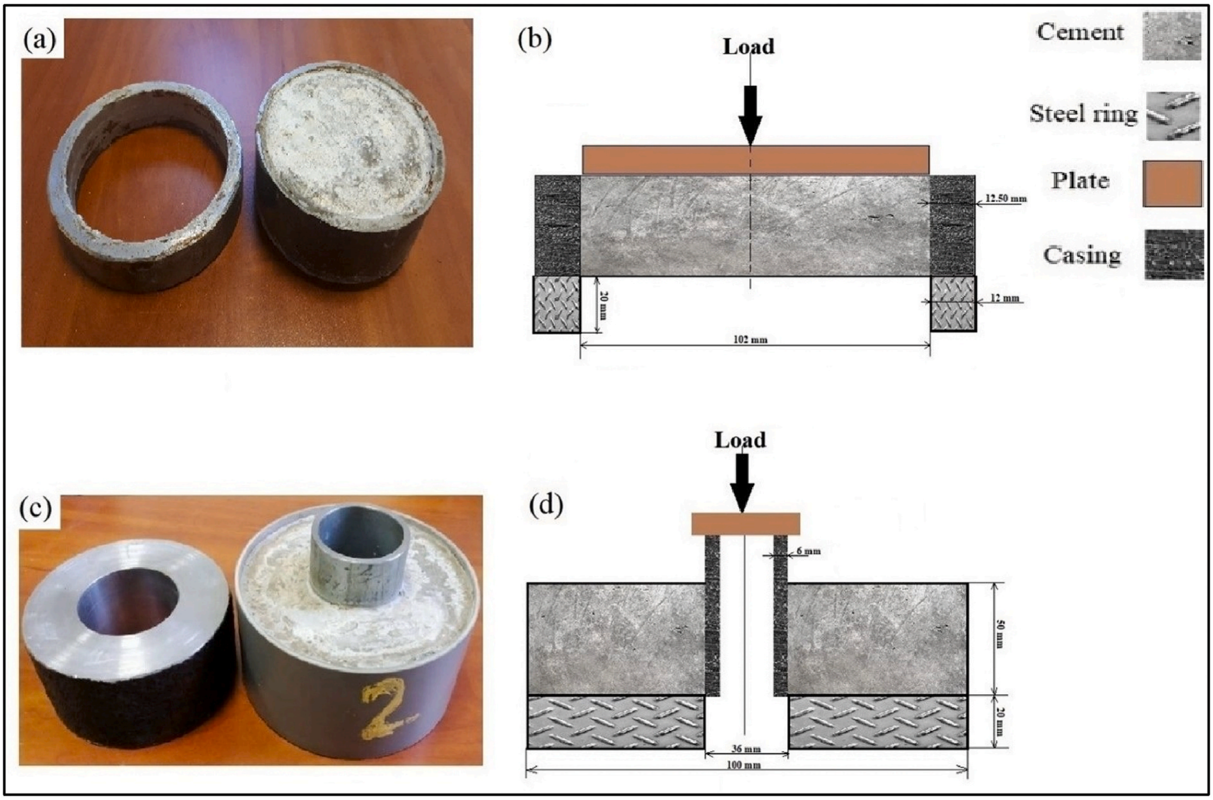

Fig. 7. Interfacial bonding shear strength in two cases: the first case (a, b) and second case (c, d).

used to measure the height and width of each sample, and the minimum surface area was recorded. The maximum axial load that the samples could withstand while in compression was then determined through testing. The axial load was applied using a compression machine with a maximum capacity of 250 kN at a steady loading rate of 18 kN/min without any vibration. For each tested age, the average test results on three samples from each concentration shall constitute the test result. After measuring the maximum axial load required to crush the cement sample, **Equation (10)** can be applied to calculate unconfined compressive strength, **Equation (11)** can be used to calculate the tensile strength [31,34,35] and **Equation (12)** can be used to calculate the flexural strength.

$$UCS = \frac{4 \times Fmax}{\pi \times D^2} \tag{10}$$

where, *UCS* is the unconfined compressive strength (MPa), *Fmax* is the maximum axial load (N), and *D* is the cement sample diameter (mm).

$$TS = \frac{2 \times F}{\pi \times D \times L} \tag{11}$$

where, TS is the tensile strength (MPa), F is the load at cement sample failure (N), D is the cement sample diameter (mm), and L is the cement sample length (mm).

$$R = \frac{3 \times F \times L}{2 \times b \times d^2} \tag{12}$$

where, R is the flexural strength (MPa), F is the load at cement sample failure (N), L is the support span length (mm), b is the cement sample width (mm), and d is the cement sample thickness (mm).

2.11. Determination of interfacial bonding shear strength

Interfacial bonding is a type of bonding that occurs between two different materials such as cement sheath and casing string. In this study, a new method is proposed to simulate the bonding between cement and casing. Also, it shows the interaction at the interface between casing couplings and the cement. Furthermore, this method demonstrates the impact of some additives such as APCP on the interfacial bonding shear strength. The axial load was applied using a compression machine with a maximum capacity of 250 kN at a

Table 3
Geometrical details of the first and second cases of shear strength samples.

Case number	The length of casing (mm)	The outer diameter of the casing (mm)	The inner diameter of the casing (mm)	The outer diameter of the cement (mm)	The length of cement (mm)
First case	50	114.50	102	102	50
Second case	70	42.33	36.40	100	50

steady loading rate of 18 kN/min without any vibration. This hydraulic press was equipped with a digital device to measure the applied load in (N). This paper was introducing laboratory tests for shear bonding failure between cement sheath and casing that are performed on two different cases as shown in Fig. 5. The first case consisted of measuring the axial load needed to push out a 20 mm of the cement plug that was previously cast into a cylinder casing and cured in the water bath for different curing time (7,14, and 28 days) at atmospheric pressure and room temperature (24 °C) conditions. This case simulates the process of closing the well with a cement plug when abandoning the well or starting to drill the dogleg section in deviated wells. Thus, there is a single contact area between the cement and the inner face of the casing as shown in a cross-sectional view in Fig. 7a and b. The plate that is used to apply the load to the cell has a circular shape and a diameter slightly smaller than the inner diameter of the casing, ensuring that no friction occurs with it. A steel ring specially designed for this experiment is placed under the sample with a height of 20 mm and an inner diameter of 104 mm (greater than the inner diameter of the casing), allowing the cement to pass through it without any friction, ensuring the accuracy of the results. The second case involved measuring the axial load needed to push out 20 mm of the metal tube that was previously cemented in the middle of the cement plug and after that the samples should be cured in the water at atmospheric pressure and room temperature (24 °C) conditions for different curing time (7,14, and 28 days). This case simulates the most common position of cement in the annular space between the casing and the formation. In this case, the single contact area is between the cement and the outer surface of the casing as shown in a cross-sectional view in Fig. 7d and c. In this case, a steel ring specially designed for this experiment is placed under the sample, with a height of 20 mm, an outer diameter of 100 mm and an inner diameter of 45 mm, allowing the casing to pass inside it without any friction, which ensures the accuracy of the results. Also, the purpose of this work is to measure the interfacial bonding shear strength of class G neat cement and to compare the results with cement containing an increased concentration of APCP to understand the effect of APCP on the interfacial bonding shear strength. In these experiments, parts of the two different diameters of a casing were used for both cases prepared from the same tube, thus it has the same specifications of dimensions and roughness without using any coating. Also, the contact surface between the cement and the casing should be dry and clean of any impurities before preparing samples. It is also important to ensure that there are no air bubbles inside the cement samples in both cases. For each tested age, the average test results on three samples from each concentration shall constitute the test result. For the sake of reliable results, the casing is only used once during the experiments and is not reused again. Table 3 shows the geometrical dimensions of the first and second cases including the length, outer diameter, and inner diameter of casing and cement. After measuring the axial load needed to push out the cement or the casing, the following **Equation (13)** is applied to calculate the interfacial bonding shear strength of both cases.

$$\sigma = \frac{Fmax}{\pi \times D \times L} \tag{13}$$

where, σ is the interfacial bonding shear strength (MPa), *Fmax* is maximum axial load used during the test (N), *D* is referring to the inner diameter of the casing for the first case and outer diameter of the casing for the second case of the casing (mm), and *L* the interfacial area length between the casing and the cement (mm).

2.12. Thermogravimetric analysis of cement samples

The thermal analysis technique by using a Derivatograph C/PC was performed to evaluate the influence of the heating on the mineralogical composition of well cement hydration products. As the cement was heated to a high temperature, it begins to decompose and lose weight. In this study, the cement samples should be cured for 28 days at the normal conditions of pressure and temperature. Next, the samples were dried in an oven for 24 h at 100 °C. The samples were then left to cool to room temperature. Then, the inner core of the sample was crushed and ground to 100 μm before being used in the test. Then, a neat cement sample and a cement sample containing 5 %BWOC of APCP were exposed to temperature up to 1200 °C and the heating rate was 10 °C/min. Changes in weight loss with elevated temperature were tracked and analyzed. The thermogravimetric analysis (TGA), and derivative thermogravimetric analysis (DTA) curves were used to evaluate the thermal degradation. They also help in the analysis of all peaks resulting from the weight losses, as well as provide the possibility to determine different parameters such as temperature with different decomposition degrees and weight loss at each stage.

3. Results and discussion

3.1. Rheological properties results

Many parameters, including the (S/W), the cement slurry homogeneity, and the interaction between cement and chemical additive,

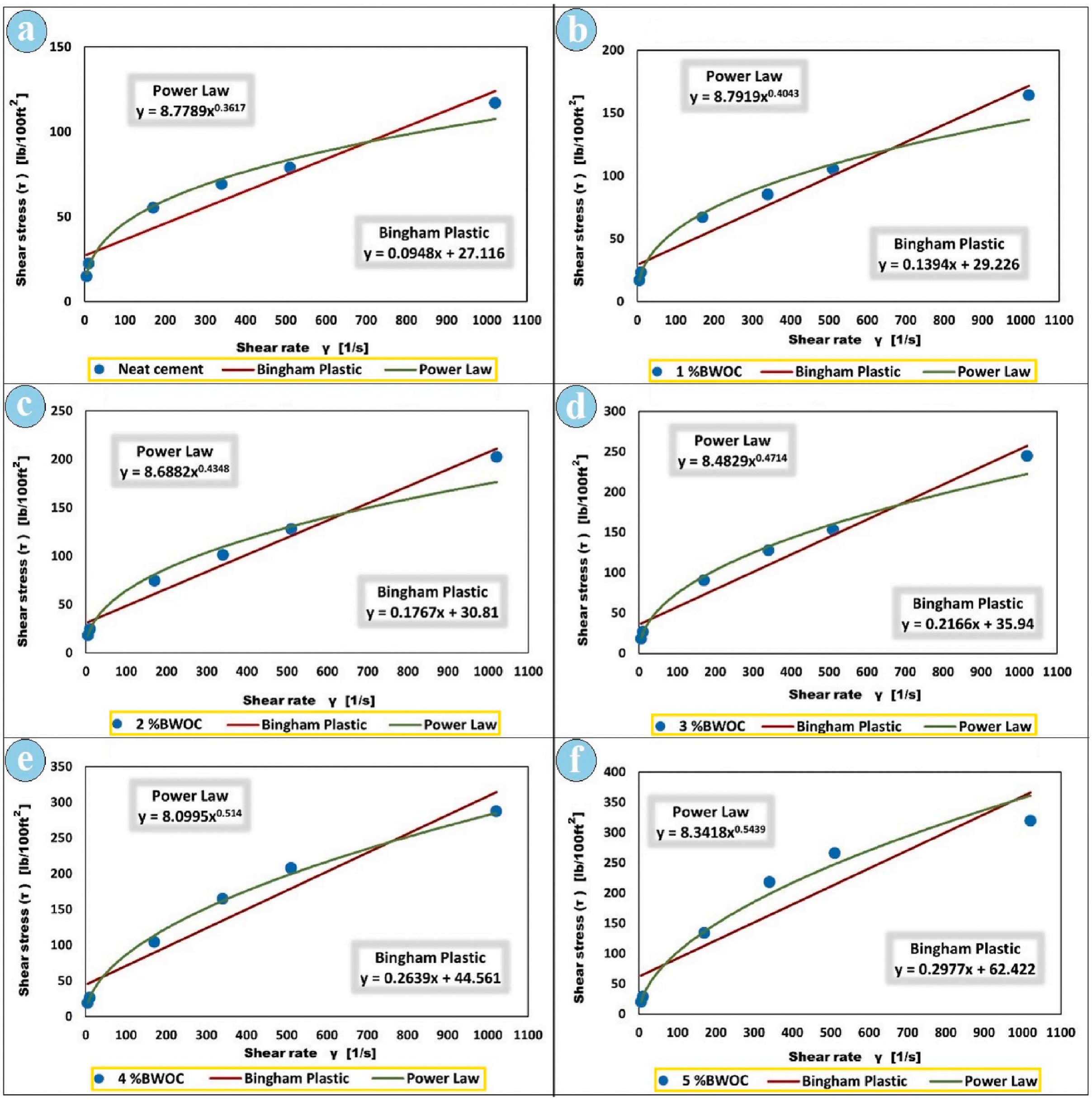

Fig. 8. Shear stress vs. shear rate for the cement samples containing (a) 0 %BWOC of APCP, (b) 1 %BWOC of APCP, (c) 2 %BWOC of APCP, (d) 3 % BWOC of APCP, (e) 4 %BWOC of APCP, and (f) 5 %BWOC of APCP.

have a significant effect on the rheological properties of the cement slurry. This section investigated the impact of adding APCP to well cement slurry with an increasing concentration on rheological parameters. Fig. 8 depicts the rheological behavior of all cement slurry systems designed for this study, which differed by the proportion of APCP added (0,1,2,3,4, and 5% BWOC, i.e., 1 g of APCP added to 100 g of cement at 1 g/100 g). The most common mathematical models employed in the sector to investigate the rheological behavior of cement slurry are the power law model, the Bingham plastic model, and the Herschel-Buckley model. Bingham plastic is the most used model due to its linear model and applicability. Power law model and Bingham Plastic model have been combined to create Herschel-Bulkley model, which takes into account the yield stress effect in the Power Law model [36,37].

From Fig. 8, by plotting shear stress versus shear rate the diagram shows two models the first one is a Bingham plastic model where the slope of the diagram represents the plastic viscosity (PV) while the intersection of the diagram represents the yield point (YP). The other model is the power-law which includes the consistency index (k) and behavior index (n) in the power-law model specifying the apparent viscosity and degree of non-Newtonian behavior in the cement slurry, respectively and Herschel-Bulkley model includes three parameters which are (n), (k), and yield stress (is the intercept point between power law and Bingham Plastic diagram). It was found that by comparison between the three rheological models (Bingham plastic, power-law, and Herschel–Bulkley) and measured

Table 4
Rheological properties of cement samples with increasing concentrations of APCP.

Sample	Herschel-Bulkley			
	Bingham Plastic		Power Law	
	PV (cp)	YP (lb/100 ft^2)	K (lbf.s^n/ft^2)	n
Slurry I	45.14	27.116	0.1834	0.3617
Slurry II	66.38	29.226	0.1836	0.4043
Slurry III	84.14	30.810	0.1815	0.4348
Slurry IV	103.14	35.940	0.1772	0.4714
Slurry V	125.67	44.561	0.1692	0.5140
Slurry VI	141.76	62.422	0.1742	0.5439

Table 5
Percentage of free water in the cement slurry at different concentrations of APCP.

Sample	Specific gravity	Free fluid volume (mL)	Free fluid percentage (%)
Slurry I	1.93	8.65	3.46
Slurry II	1.90	5.20	2.08
Slurry III	1.88	2.10	0.84
Slurry IV	1.87	1.00	0.40
Slurry V	1.84	0.00	0.00
Slurry VI	1.83	0.00	0.00
API Limit	-	-	**5.90**

Table 6
Sedimentation test results of cement samples with increasing concentrations of APCP.

Sample	Sedimentation Stability							
	Density in the column parts (Top part to bottom part) (g/cm^3)						$\Delta\ \rho$ (g/cm^3)	Downgrades (mm)
	Top Part	Part 1	Part 2	Part 3	Part 4	Bottom Part		
Slurry I	1.936	1.940	1.950	1.956	1.968	1.984	0.048	3.20
Slurry II	1.921	1.936	1.944	1.953	1.957	1.962	0.041	2.42
Slurry III	1.909	1.918	1.927	1.932	1.939	1.943	0.034	1.18
Slurry IV	1.904	1.909	1.915	1.918	1.921	1.925	0.021	0.91
Slurry V	1.887	1.888	1.887	1.889	1.901	1.901	0.014	0.64
Slurry VI	1.861	1.862	1.862	1.863	1.869	1.871	0.010	0.45
API Limits							**0.060**	**5.00**

data that the power-law model is the most appropriate model to describe the behavior of cement after adding the APCP, with an average absolute percentage error (AAPE) that did not exceed 0.077.

The plastic viscosity (PV) and yield point have increased as reported in Table 4 due to the increase in APCP concentration from 0% BWOC to 5%BWOC (YP). The neat cement slurry without containing any APCP additive showed the lowest plastic viscosity (PV) and the yield point (YP) values compared to cement slurry containing an increasing concentration of APCP. The 5 %BWOC dosage of APCP increase the plastic viscosity and yield point by 3.14 and 2.30 times higher than the neat cement slurry respectively. It can be said that as the APCP concentration increased, so did the rheological parameters. This increase is attributable to an increase in the ratio of solid content to liquid content in the cement slurry. It results in difficulties in pumping the cement slurry during cementing job due to increasing the frictional loss. According to recent studies, an increase in the solid concentration leads to an increase in the rheology of cement-based materials, an increase in interlayer friction and a high-density filler for cement construction [38–40]. However, the values of the rheological parameters are within the acceptable range for well cementing.

3.2. Slurry stability results

In laboratory tests, free water is recorded by volume but calculated as a percentage of cement slurry. The results obtained from the free water test are given in Table 5. As can be seen from the data in Table 5, the volume of free water decreases as the concentration of APCP in the cement slurry increases. It can also be observed from Table 5 that with the addition of 4 BWOC% and 5 BWOC% of APCP, no free water was recorded on the top of the cement slurry. This indicates the efficiency of the APCP in reducing the volume of separated water and holding the free water in the dispersion system of cement. This greatly helps in maintaining the original properties of the cement slurry and prevent influx on the top part of the cement while it is setting in the well. It is reducing the possibility of channels forming within the cement during the cementing job due to the decrease in hydrostatic pressure caused by the decrease in effective density with the increase in the amount of free water [41]. This decreases attributable to an increase in the ratio of solid

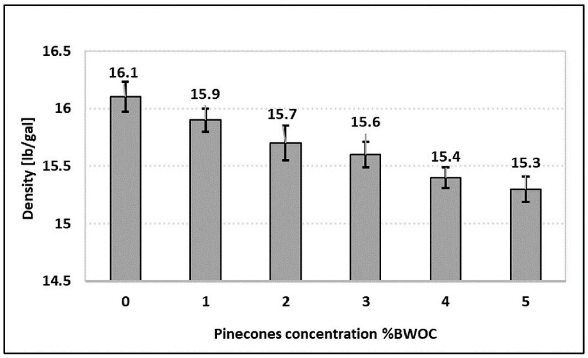

Fig. 9. The density of cement slurry at different concentrations of APCP.

content to liquid content in the cement slurry and the fact that APCP has a hydrophilic nature since it is a cellulosic material. That help to formation of the homogeneous cement slurry, which provides good zonal isolation in the oil well. The same results were found in The research conducted by Ref. [42] showed that an increase in the corn husk Ash concentration (up to 5%) led to a decrease in the amount of free fluid that has accumulated on top of the formed slurries. Nevertheless, none of the cement systems utilized in this study exceeded the API 10-A maximum limit, which ensures that the proportion of free water in the cement slurry does not exceed 5.90%. It should be noted that the minimum volume of free water is mostly determined by the cement slurry viscosity used. As a result, and as mentioned earlier, the addition of APCP had a significant impact on increasing the viscosity of the cement slurry which affected the volume of separated free water.

Table 6 shows the results of the sedimentation test on cement samples containing increased concentration of APCP. The density of the neat cement sample in the top part of the column is 1.936 g/cm3 and in the bottom part 1.984 g/cm3. This indicates that light microscopic particles floated at the top of the cement column, while heavy particles settled at the bottom, resulting in a density difference of 0.048 between the two ends. As can be seen in the table, the situation changes as the concentration of APCP increases. The density difference between the various parts of the cement column decreases gradually and the density tends to be equal in the different parts as the APCP concentration increases. This can be the result of the APCP particles filling the pores and enhancing water absorption. It has been proven by experience that pine cones absorb water greatly due to the absorption property of their cells, which increases their volume as a result of swelling of the amorphous components (hemicellulose) [43]. Therefore, APCP can be used as fillers for voids inside the cement sheath since it helps to prevent particles from moving up or down due to density differences. The difference in sample length before and after curing for 24 h is referred to as (Downgrades). It can be seen from the table that the addition of APCP led to a slight decrease in the length of the sample compared to the neat cement sample. However, the density differences and downgrades in this study did not exceed the limit determined by the American Petroleum Institute (API).

3.3. Density test results

The mass per unit volume can be used to determine the density. The Model 140 Fann Mud Balance was used to measure the density of the cement slurry. Fig. 9 presents the results of the cement density test. The density of the cement slurry decreases slightly as the concentration of APCP increases by compared to neat cement. These results are consistent with the results of [44,45], where there was a decrease in the density with the increase in the concentration of agricultural waste and cellulosic materials in well cement slurries. Also, it's worth noting according to previous research, that using natural fibers in cement-based materials reduces the density of the cement [46–49]. In the case that 5% BWOC of the APCP was added to cement slurry, the density value decreased by approximately 5% compared to neat cement slurry. This can be attributed to the lightweight particles of APCP with specific gravity equal to (0.4788), which could result in reducing the density of cement slurry when mixed in increasing concentration with class G cement which has a specific gravity (3.15) [35]. However, the density distribution is more even. According to the results, APCP can be employed as a cement extender to reduce the hydrostatic pressure of the cement column against formation zones where lost circulation can occur. Practically, the density of the cement slurry used in a well cementing job varies between 11.5 (lb/gal) to 19.0 (lb/gal) [50].

3.4. Fluid loss test results

Fluid loss is one of the common problems for the oil and gas industry during the drilling operation. This test simulates the cement pumping and setting process within the well. If there is a large volume of fluid loss led to a reduction in the quality of cementing job [51]. In this case, The gas has the potential to enter the annulus where the cement slurry is to be placed and create channels that lead to losing the insulating function of the cement sheath in the well [52]. The fluid loss of the cement slurry with increasing concentration of APCP was measured at the end of 30 min, as shown in Fig. 10. The fluids loss of the cement slurry shows a declining trend with the addition of increasing concentrations of APCP. From the results, the fluid loss shows a decrease by 37% for 5 %BWOC of APCP. This decrease may be due to the absorption of water by the APCP particles trapped within the cement slurry and creating a filter cake with low permeability to keep the fluid in the cement slurry from leaking into the formation. It is interesting to note that the fluid loss was

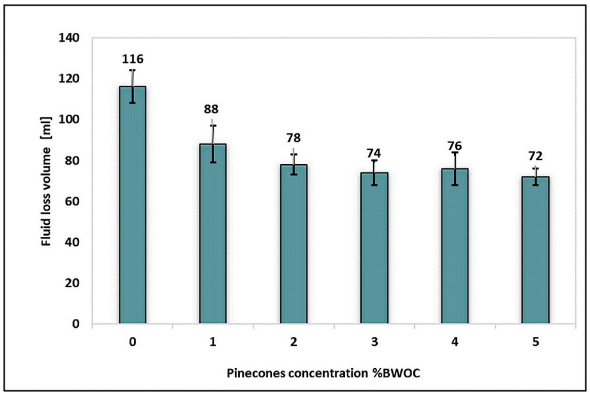

Fig. 10. API fluid loss volume of the cement slurry at different concentrations of APCP.

Table 7
Results of porosity and permeability measurements of cement samples containing APCP.

APCP (% BWOC)	Diameter of sample (cm)	Length of sample (cm)	Mass of sample (g)	Porosity value of sample (%)	Average solid volume of sample (cm^3)	Permeability of sample (mD)
0	3.606	5.695	91.80	32.03	39.53	0.186
1	3.612	5.128	57.20	52.60	24.91	0.175
2	3.601	5.276	60.65	50.95	26.36	0.116
3	3.598	5.368	63.64	49.23	27.71	0.145
4	3.586	5.654	62.60	52.16	27.32	0.163
5	3.593	5.437	82.09	35.70	35.44	0.088

significantly decreased after adding 1%BWOC of the APCP and with increasing APCP concentration the rate of decline has decreased. As a result, it is clear to say that adding APCP play an important role in retaining the chemical and physical properties of cement slurry and resulting in a successful cement job. Therefore, APCP can be used as a fluid loss control agent for the cement slurry against porous formations, preventing early dehydration of the cement slurry before the scheduled time. Cellulosic materials are known to be one of the most common fluid loss additives. However, cellulose polymers increase the time required for cement hardening and increase its viscosity. In the study conducted by Refs. [53,54], in which it was proven that adding cellulosic materials like hydroxyethyl cellulose, carboxymethylcellulose and hydroxypropyl methylcellulose to well cement contributes to reducing fluid loss. The study performed by Ref. [55] proved that sugarcane fiber helped reduce the loss of circulation, density and free water volume. In oil wells, For oil wells, the maximum permissible fluid loss rate is 200 mL per 30 min, and for gas wells, it is 50 mL per 30 min [56,57]. In light of this expression, the cement slurry in this study did not exceed the permissible limit for the oil well. APCP can be considered an eco-friendly material that would be an ideal fluid loss additive.

3.5. Porosity and permeability tests results

Table 7 displays how the addition of different APCP concentrations to the cement samples changed the porosity and permeability values. The porosity of the neat cement sample was 32.03%. It is clear to observe from the results that the porosity increased to become 52.60%, 50.95%, 49.23%, and 52.16% when adding the APCP at a concentration of 1 %BWOC, 2 %BWOC, 3 %BWOC, and 4 %BWOC, respectively. But with the addition of 5 %BWOC of APCP, the porosity was decreased to 35.70%. Fig. 11 shows the ratio of solid volume to void volume (Vs/Vp) for cement samples with increasing concentrations of APCP.

Regarding permeability, the table presents the change in permeability values of cement samples containing different concentrations of APCP. It is clear to say that the permeability values have decreased with the increase in the concentration of APCP. The permeability of the neat cement sample was 0.186 mD. It is clear to observe from the results that the permeability decreased to become 0.175 mD, 0.116 mD, 0.145 mD, 0.163 mD, and 0.088 mD when adding the APCP at a concentration of (1, 2, 3, 4, and 5) %BWOC, respectively. The decrease in the permeability is due to some pores being filled with the APCP particles. This indicates that the APCP can be used as a filler within the formation of a densified cement sheath due to its permeability-reducing property. The variability in permeability for nitrogen as a function of mean pressure is seen in Fig. 12. According to the study (Anjos et al., 2013), adding sugarcane biomass waste to well cement at increasing concentrations reduced the permeability of the cement samples. Generally, the permeability of well cement is considered acceptable if it is less than 0.1 mD.

3.6. Water absorption, and bulk density test results

This type of test is not familiar for well cement, but it was performed to confirm the results of other tests and to verify the influence

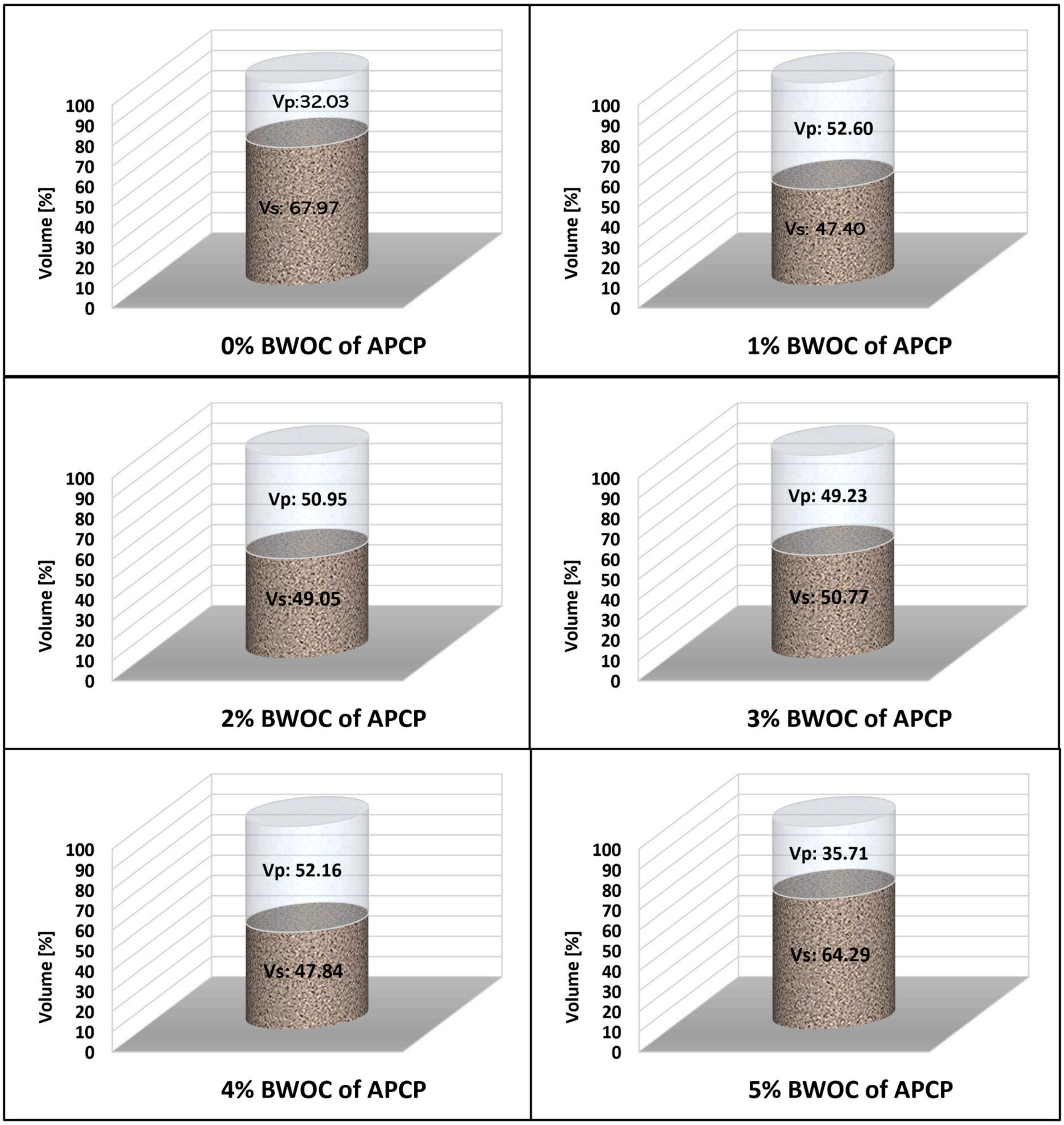

Fig. 11. The ratio of solid volume (vs) to void volume (vp) for solid cement samples after adding (a) 0 %BWOC of APCP, (b) 1 %BWOC of APCP, (c) 2 %BWOC of APCP, (d) 3 %BWOC of APCP, (e) 4 %BWOC of APCP, and (f) 5 %BWOC of APCP.

of the APCP on the improvement of the water absorption property. In oil wells, there are in direct contact between the cement sheath and formation fluids, particularly water. As a result, water absorption is a critical factor in the cement sheath's integrity. To the best of our knowledge, this property has never been addressed in an oil well cement research study. This test also helps to understand how the addition of APCP affects the bulk density of the cement sheath. In Fig. 13, the results showed that adding increasing APCP concentrations increased water absorption. This makes the behavior of well cement with APCP a complex problem, due to two reasons: First, because the APCP absorbs a lot of water, there may not be enough water in the cement system to start the cement hydration reactions [58]. The high water absorption leads to the accumulation of water inside the cement which creates a weak point in the cement [59, 60]. If these natural fibers are added to the cement matrix, the mechanical properties of the cement deteriorate as a result of this problem. The second reason is that if these natural fibers do not absorb water well, another problem may occur which is bad adhesion between these fibers and cement particles [61,62]. Water absorption is well recognized to be closely related to porosity, particularly open pores that are connected to the surface of the sample since water generally passes via the pores of the samples. The lowest value of

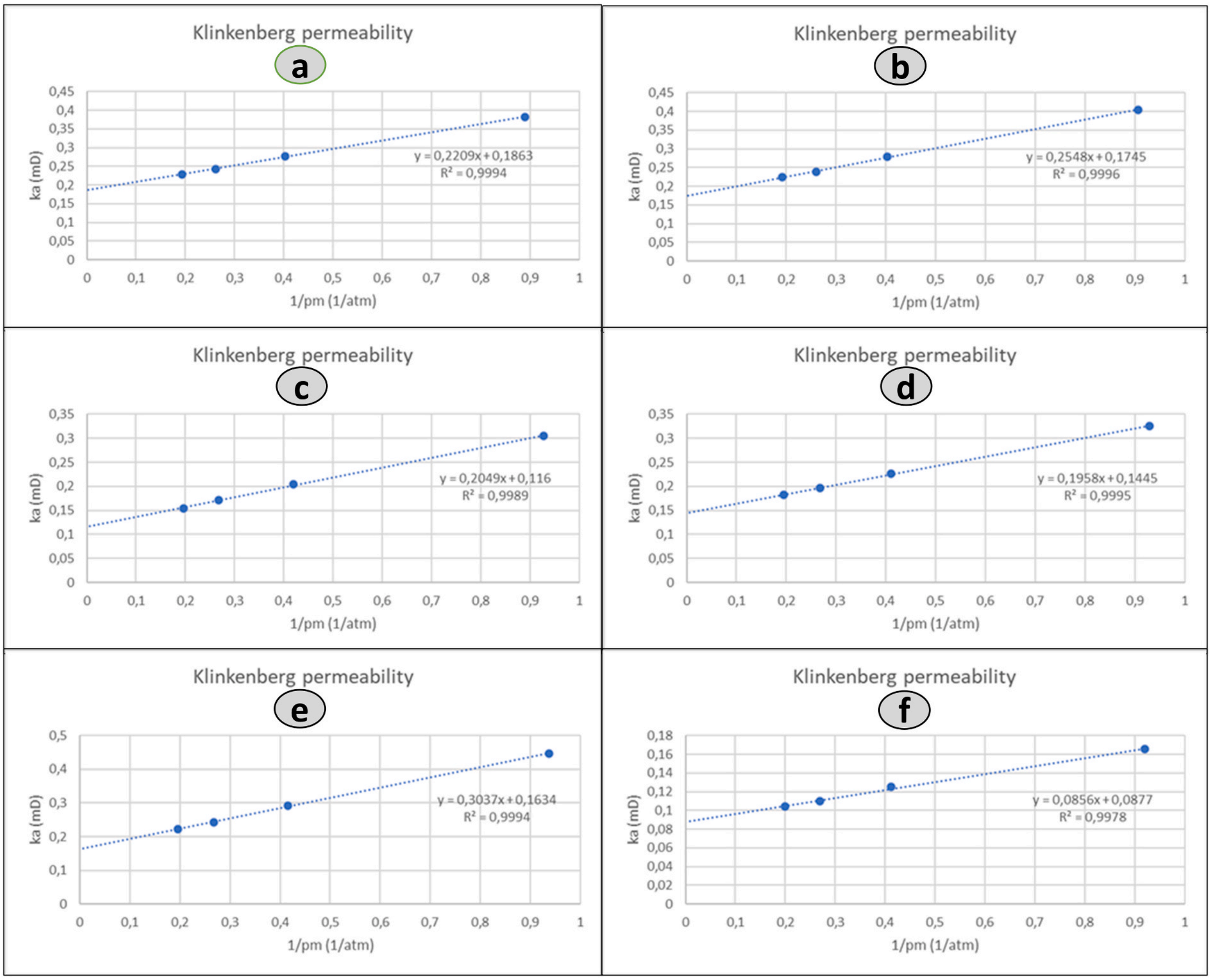

Fig. 12. The change in permeability as a function of mean nitrogen pressure for solid cement samples after adding (a) 0 %BWOC of APCP, (b) 1 %BWOC of APCP, (c) 2 %BWOC of APCP, (d) 3 %BWOC of APCP, (e) 4 %BWOC of APCP, and (f) 5 %BWOC of APCP.

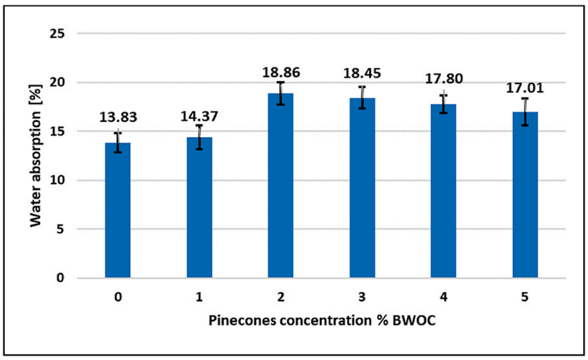

Fig. 13. Water absorption results with the addition of increasing concentrations of APCP.

water absorption was recorded to be 13.83%, obtained from the neat cement sample. In contrast, the cement sample with 2 %BWOC of APCP showed a high absorption value of 18.86%. It should be noted that pine cones absorb water. The bulk density has decreased significantly by adding increasing concentrations of APCP to the cement matrix, which provides a lighter and more insulating cement sheath as can be seen in Fig. 14. Based on the previous discussion, it has been found that employing natural fibers in cement without prior treatment is practically useless.

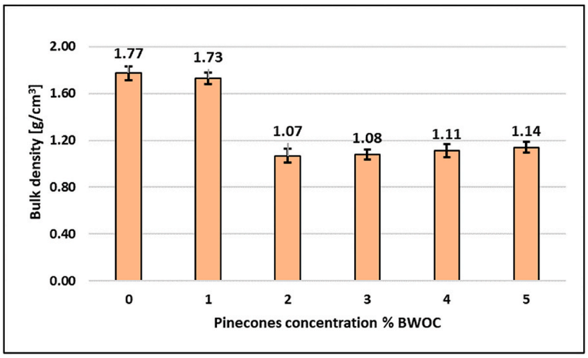

Fig. 14. Bulk density results with the addition of increasing concentrations of APCP.

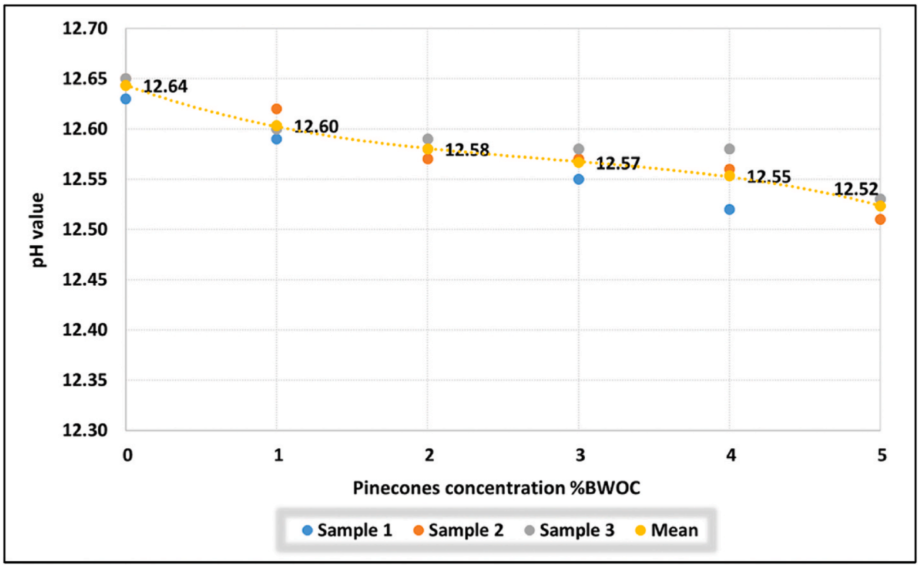

Fig. 15. pH values of the cement samples at different concentrations of APCP.

3.7. pH test results

Well cement should be extremely alkaline to protect the well casing from corrosion and improve well integrity. The high pH of well cement provides a passive protective coating on the casing. The pH measurement was carried out on three solutions of each concentration and the average value was calculated. Fig. 15 depicts the results of the pH measurement after adding increasing concentrations of APCP. The results show a slight decrease in the pH value with increasing the concentration of the APCP. Neat cement samples have average pH of 12.64 which was the highest value compared to other samples containing APCP. Through these results, it can be concluded that adding APCP to cement has no significant impact on its alkalinity.

3.8. Unconfined compressive strength (UCS), tensile strength (TS), and flexural strength tests results

Fig. 16 depicts the relationship between the compressive strength and concentrations of APCP at different aging times. Neat cement samples have average compressive strengths of 18.21 MPa, 19.88 MPa and 28.55 MPa after aging for 7, 14 and 28 days, respectively. The compressive strength of cement samples is reduced by the addition of APCP. At each of the three ages, different changes are noticed on compressive strength compared to neat cement samples. It is also worth noting that the compressive strength of the cement samples at early ages 7 and 14 days is very low compared to those at later ages 28 days. This may be due to the fact that the transition from water and cement components to Calcium Silicate Hydrate (C–S–H) gel begins on the first day after mixing and takes 28 days to complete as reported by Ref. [63]. Based on the results of this study, the compressive strength of cement samples at different curing times is adversely influenced by an increase in APCP concentration. Usually, when cement additives are used, the compressive strength decreases, regardless of the kind and concentration of these additives due to reducing the C–S–H and Portlandite concentrations in the cement matrix as well as disruption of the interlocking mechanism from giving the strength [64,65]. Thus, it is likely that the addition

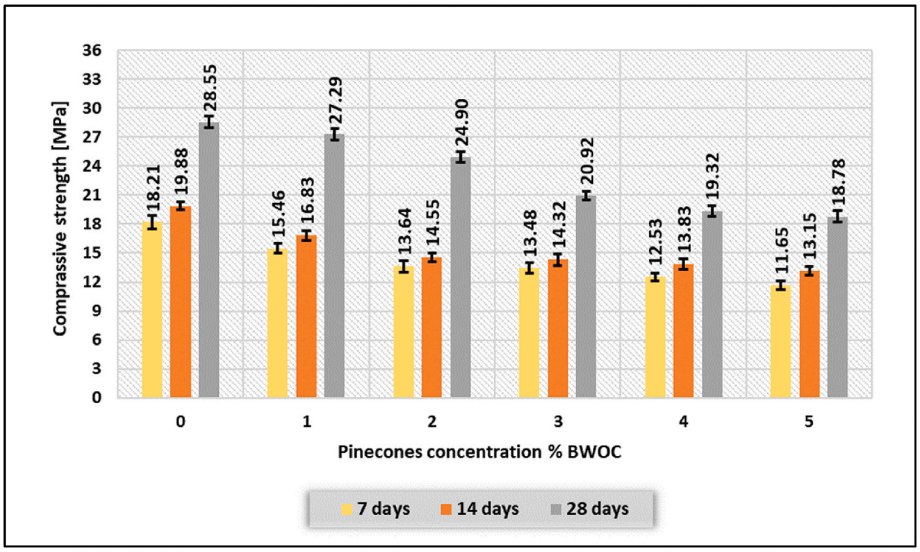

Fig. 16. Unconfined compressive strength (UCS) of the cement samples at different concentrations findings for compressive strength, of APCP.

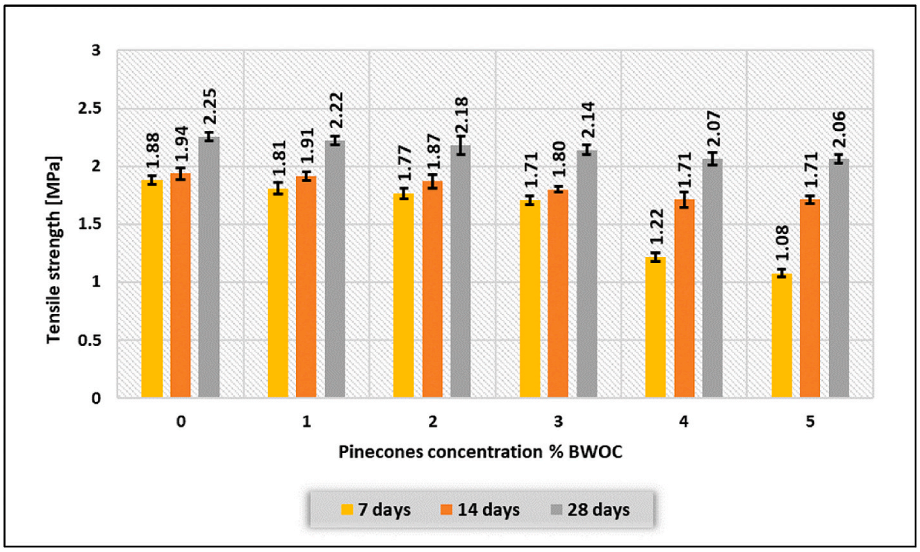

Fig. 17. Tensile strength (TS) of the cement samples at different concentrations of APCP.

of APCP is responsible for the decrease in compressive strength. This was aligned with the findings obtained by Ref. [66], who found that adding waste of Aleppo pine wood to concrete reduces the compressive strength value. The use of natural pozzolan in the study presented by Ref. [67] also led to a significant decrease in the compressive strength with an increase in the concentration of pozzolan. This means that additives of different types and concentrations negatively affect the compressive strength of cement samples. This also can be explained by the result of porosity measurement. It can be observed that the porosity of the samples increased after the addition of APCP, which makes the cement samples more brittle compared to the neat cement samples due to the formation of a porous structure. The presence of pores is explained by the amount of water absorbed by the APCP particles, which increased with the increase in the concentration of the APCP, as mentioned previously. However, based on the results, all of the samples had compressive strengths that were higher than the minimum requirements set forth by API standers for well cement. It was found thatadding APCP up to 5% BWOC can result in a cement with a compressive strength of 18.78 MPa, which is an acceptable value for well cement according to API 10A standards, which specify a minimum range of compressive strength is 2.06–10.34 MPa [24].

According to the findings shown in Fig. 17 indicate that the tensile strength decrease as the concentration of the APCP increases. Neat cement samples have average tensile strengths of 1.88 MPa, 1.94 MPa and 2.25 MPa after aging for 7, 14 and 28 days, respectively. The tensile strength gradually decreases when the APCP adding percentages range from 1 %BWOC to 5 %BWOC. At all aging times, the tensile strength of the neat cement sample is the largest and that of cement slurries with 5 %BWOC of APCP is the

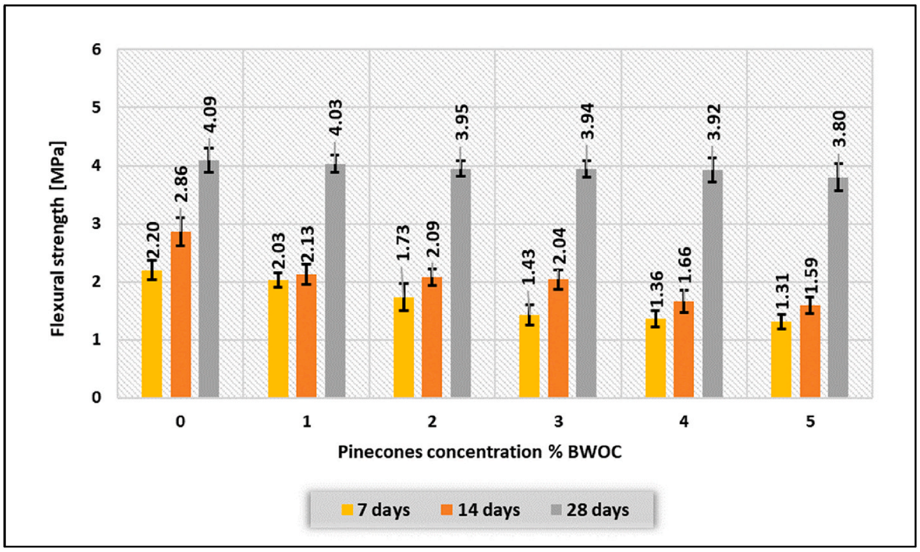

Fig. 18. Flexural strength of the cement samples at different concentrations of APCP.

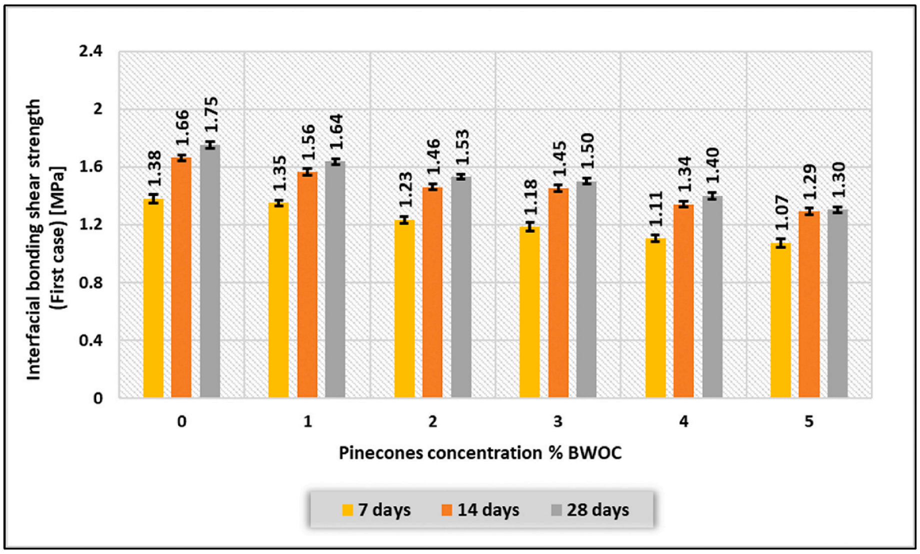

Fig. 19. Interfacial bonding shear strength of the cement samples at different concentrations of APCP for first case.

smallest. After 7 days of aging, the tensile strength decreased by 3.65%, 5.97%, 9.01%, 35.20% and 42.63% when the APCP concentration is 1 %BWOC, 2 %BWOC, 3 %BWOC, 4 %BWOC and 5 %BWOC, respectively. After 14 days of aging, the tensile strength decreased by 1.18%, 3.42%, 7.15%, 11.64% and 11.57% when the APCP concentration is 1 %BWOC, 2 %BWOC,3 %BWOC,4 %BWOC and 5 %BWOC, respectively. After 28 days of aging, the tensile strength decreased by 1.46%, 3.33%, 5.08%, 8.36% and 8.53% when the APCP concentration is 1 %BWOC, 2 %BWOC,3 %BWOC,4 %BWOC, and 5 %BWOC, respectively. It is interesting to note that the rate of decrease in tensile strength of samples after curing for 28 days is much less than the rate of decrease in tensile strength of samples after curing for 7 and 14 days. In general, a slight decrease in tensile strength was observed with the increasing concentration of APCP. Similar to the increasing APCP concentration reduced the amount of stable C–S–H formation in the cement matrix, which led to the deterioration of the tensile strength. According to the study conducted by Ref. [68], adding sugarcane bagasse ash to concrete reduced tensile strength.

Fig. 18 shows the results of the flexural strength test. Generally, the flexural strength increases as the aging time increases. As can be seen from the data, the flexural strength of cement samples reached the maximum when the concentration of APCP is 0 %BWOC (Neat cement sample) and tends to decrease when the concentration of APCP is increased from 1%BWOC to 5%BWOC. Neat cement samples have average flexural strengths of 2.20 MPa, 2.86 MPa and 4.09 MPa after aging for 7, 14 and 28 days, respectively. The flexural strength gradually decreases when the APCP adding percentages range from 1 %BWOC to 5 %BWOC. At each of the three ages,

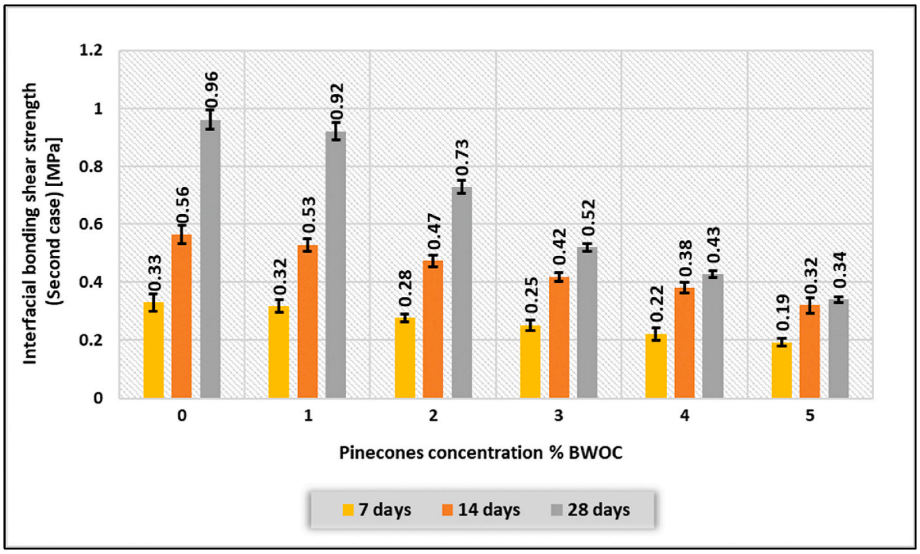

Fig. 20. Interfacial bonding shear strength of the cement samples at different concentrations of APCP for second case.

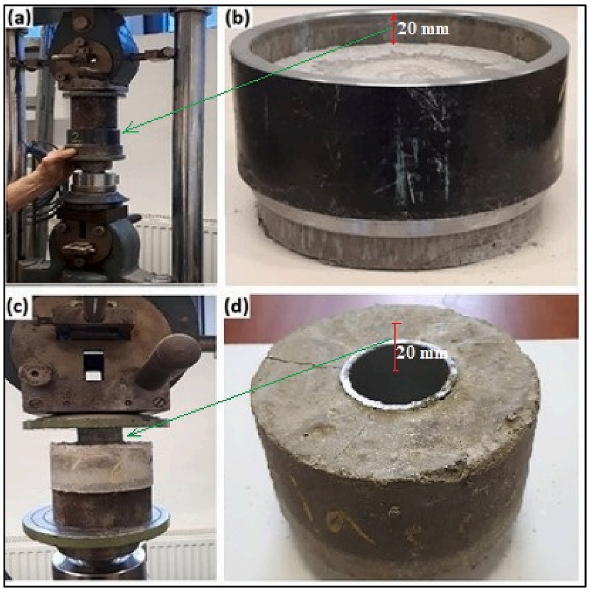

Fig. 21. Interfacial bonding shear strength sample for the first case (a)during and (b) after testing, and for the second case (c)during and (d) after testing.

different changes were noticed on flexural strength compared to neat cement samples. However, regardless of concentration, the flexural strength of cement containing APCP remained extremely near to the values at 28 days of age when compared to the neat cement sample. The smallest value of flexural strength was 3.80 MPa obtained at 5%BWOC concentration, which is the maximum concentration of APCP used in this study. These findings are consistent with those made by Ref. [69] who found that adding guinea corn husk ash to cement leads to a decrease in the flexural strength.

3.9. Interfacial bonding shear strength test results

Fig. 19 represent the results of the interfacial bonding shear strength test for the first case between the cement and the inner face of the casing sample. The results of this test are consistent with those obtained from previous tensile, flexural, and compressive strength testing. The interfacial bonding shear strength reached the maximum when the concentration of APCP is 0 %BWOC (Neat cement sample) and tends to decrease when the concentration of APCP is increased from 1%BWOC to 5%BWOC. Neat cement samples have

Table 8
A summary of recent research related to measuring the interfacial bonding shear strength, with comparisons to the results of this study.

Author	[70]	[71]	[72]	[73]	[74]	[75]	This work
Case descriptions	Cement plug inside the casing	Casing in the middle and cement around it.	Casing in the middle and cement around it	Casing in the middle and cement around it	Casing in the middle and cement around it	Cement plug inside the casing	Cement plug inside the casing and Casing in the middle and cement around it
Interfacial bonding shear strength [MPa] (Aging time)	6.30 (7 days), 10.40 (7 days), 12.30 (14 days)	0.56 (no data)	0.10–1.00 (no data)	2.93–7.46 (5 days)	1.47–2.41 (3 days)	0.47 (1 day) −10.40 (82 days)	**First case**: 1.07–1.38 (7 days) 1.29–1.66 (14 days) 1.30–1.75 (28 days) **Second case:** 0.19–0.33 (7 days) 0.32–0.56 (14 days) 0.34–0.96 (28 days)
Cement class and additives	Cement class H without additives	Cement class H	Cement class H	Cement class G and using sand on the interfacial surface	Cement class G	Cement class H without additives	Cement class G without and with adding APCP

average values of 1.38 MPa, 1.66 MPa and 1.75 MPa of interfacial bonding shear strength after aging for 7, 14 and 28 days, respectively. Different changes in interfacial bonding shear strength are observed at each of the three ages when compared to neat cement samples. At early ages (7, 14 days) shows small values of the interfacial bonding shear strength compared to later ages (28 days). The reason for this, similar to the compressive strength test results, could be the incomplete production of C–S–H, which is responsible for the hardness of cement and takes 28 days to complete. The same is true for the second case, as shown in Fig. 20. The interfacial bonding shear strength reached the maximum in neat cement sample 0.33 MPa, 0.56 MPa and 0.96 MPa after aging for 7, 14 and 28 days, respectively and tends to decrease when the concentration of APCP is increased. The interfacial bonding shear strength for the second case also showed lower values in the early ages compared to the following ages. This means that in both cases the interfacial bonding shear strength between the cement and the casing deteriorated with the addition of APCP. It can be seen from Figs. 17 and 18, that the bonding strength did not increase significantly with the increase in aging time after 14 days when increasing concentrations of APCP were added in both cases and the value tended to be stable with increasing aging time. This indicates that the shear bonding strength is significantly affected by the type and concentration of the additives to the well cement, as the addition of these materials hinders the cement hydration process. This indicates a strong relationship between the interfacial bonding shear strength and grade of cement hydration which helps for a deep understanding of well integrity and long-term well behavior. The interfacial bonding shear strength samples for both cases before testing are depicted in Fig. 21a and 21c and after testing are depicted in Fig. 21b and 21d.

Table 8 summarizes the results of several research papers that have been published in the literature related to interfacial bonding shear strength to check whether the data obtained in this study are comparable with other published results. However, an actual comparison with these studies cannot be made due to the inconsistent parameters such as different measuring conditions (Pressure, Temperature), casing and cement dimensions, roughness, curing time, cement class, types of additives, concentration … etc. When using the same class of cement and the same curing time, the shear strength values measured in the literature are comparable with the results of this study.

3.10. Thermogravimetric analysis results

The thermogravimetric test results for the neat cement sample and cement sample containing 5 %BWOC of APCP are presented in Fig. 22a and 22b, respectively. The neat cement sample shows two significant weight losses, as indicated by the (DTA) curve. The first weight loss occurs at 120.2 °C because of free water evaporation and C–S–H dehydration. In this case, the percentage of weight loss is 8.33%. The dehydroxylation of portlandite, and other hydration products, causes the second significant weight loss, which occurs at 490.7 °C. The percentage of this weight loss in this case is 19.52%. Furthermore, when the decarbonisation reaction is considered, the results indicate that the neat cement sample heated to temperatures above 500 °C does not result in a third weight loss. In the case of the sample containing 5 %BWOC of APCP, the results showed three main weight losses as shown by the DTA curve. As in the case of the neat cement sample, the first loss in the weight occurs at 122.7 °C was the result of the evaporation of free water and C–S–H dehydration. In this case, the percentage of weight loss is 7.48%. This percentage of weight loss is slightly less than the percentage of weight loss in the case of elegant cement at the same temperature. At 498.4 °C, the dehydroxylation of portlandite and other hydration products as well as because of the combustion of organic materials such as APCP at this temperature causes the second significant weight loss. This weight loss percentage in this case is 19.02%. This percentage is also slightly less than the percentage of weight loss for the neat cement sample at the same temperature. In addition, it is observed from the curve that there is a third weight loss appears at 780.2 °C. The percentage of the third weight loss is 23.41%. This weight loss is due to the decarbonisation of calcium carbonate present in the clinker. A total weight loss of 23.9% was observed when the neat cement sample was heated to 1200 °C as shown by the TG curve. Whereas in the case of the cement sample containing 5 %BWOC of APCP, a total weight loss of 26.6% occurs when the sample is heated to 1200 °C, which means that the addition of the APCP has contributed to an increase in the total weight loss by 2.7%.

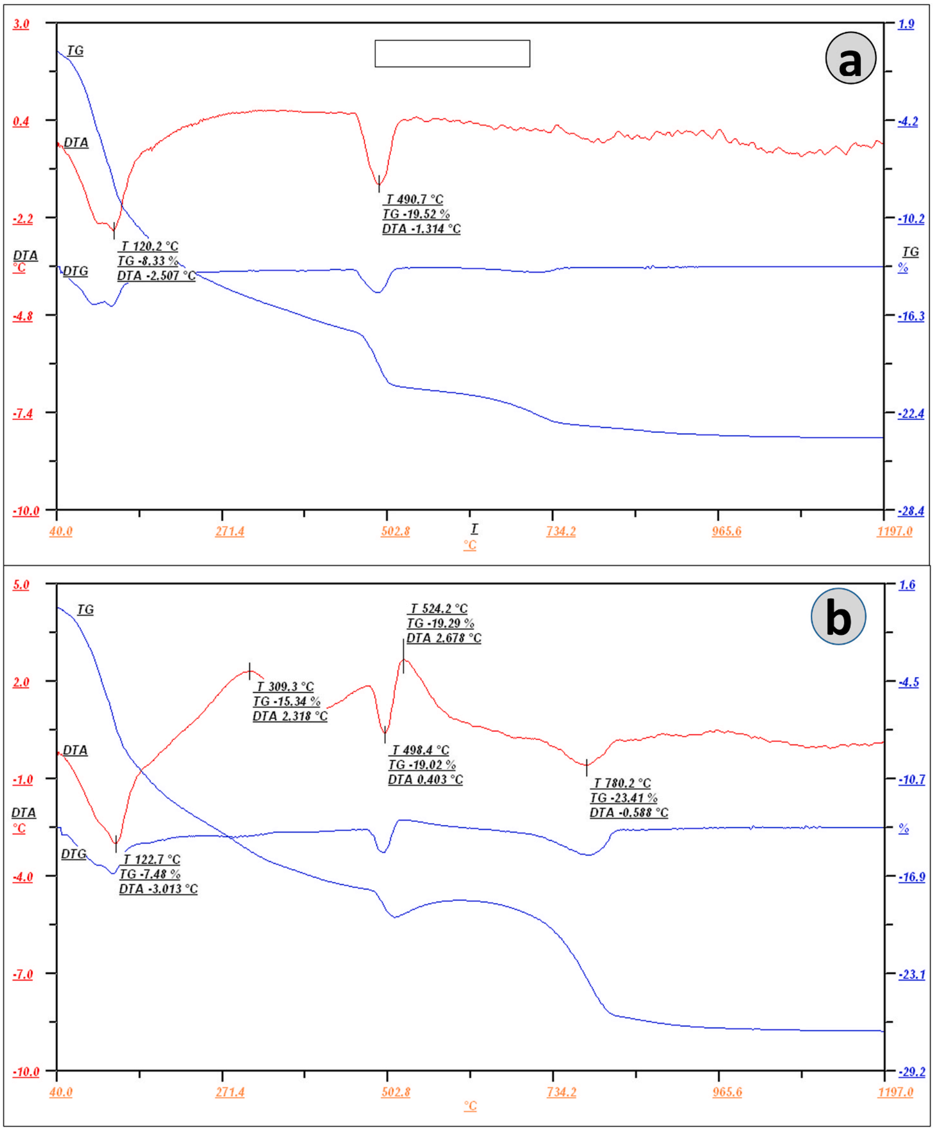

Fig. 22. TG/DTA curves of (a) neat cement and (b) cement containing 5 %BWOC of APCP.

3.11. SEM images of the APCP and pores distribution within the cement sample

Fig. 23 shows the SEM pictures of pores and APCP particles distribution within the broken cement sample resulting from the compressive strength test after being cured for 28 days in water bath. The pores appear in the form of bubbles and the APCP particles appear in black or dark colour. Crystals of calcium silicate hydrate (C–S–H) can also be seen as a bright white colour. It can be observed that the APCP was well dispersed inside the cement matrix. This good distribution prevents the occurrence of weak points in the cement sample. In terms of porosity, the samples containing the APCP had a higher porosity than the neat cement sample. However, the APCP particles contributed to the decrease in the permeability of the samples, as is evident from the permeability measurement results. The basis for improving mechanical properties is effective interfacial adhesion between the additives and the cement matrix.

4. Conclusions

In this experimental investigation, the rheological parameters, density, free water, water absorption, porosity, permeability, the volume of fluid loss, pH, mechanical properties, and thermogravimetric analysis were investigated in detail for neat Portland well cement class G and Portland well cement class G reinforced by Austrian pine cones powder (APCP). After conducting the experiments, the following main conclusions can be summarized as follows.

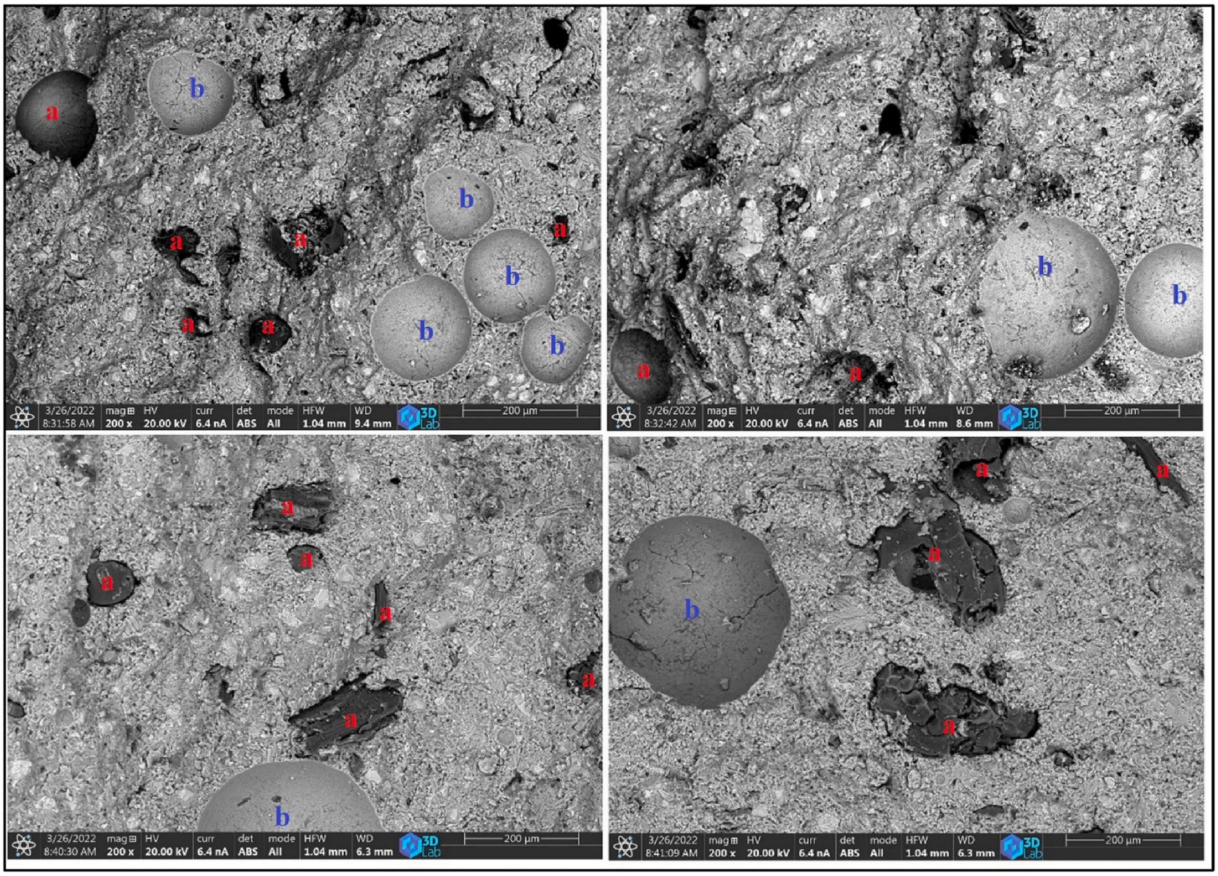

Fig. 23. SEM images of the broken face of the solid cement sample shown (a) APCP particles, and (b) pores.

- The rheological parameters of the well cement slurry containing APCP are higher than those of the neat well cement slurry.
- The addition of the APCP led to a decrease in the values of density, free water, sedimentation, and fluid loss compared to neat well cement slurry.
- The permeability values have decreased as the concentration of APCP has increased due to some pores being filled with the APCP particles. So, APCP can be used as a filler within the formation of a densified cement matrix due to its permeability-reducing property.
- In general, the addition of APCP led to a decrease in the mechanical properties of cement samples, but within the acceptable limits according to the American Petroleum Institute standers.
- The addition of APCP led to a slight increase in mass loss when exposed to high temperatures.

The findings showed that APCP, a widely accessible and inexpensive agricultural waste, does not provide cement with a high level of strength but can be considered as a new filler for well cement due to its ability to fill the pores in the cement matrix and APCP significantly reduces sedimentation effects. It can also be used as a thinner and fluid loss control agent for cement slurry.

Author contribution statement

Hani AL Khalaf: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.
Gabriella Federer Kovacsne: Conceived and designed the experiments; Performed the experiments.
Nagham Al Haj Mohammed: Performed the experiments; Analyzed and interpreted the data.
Gábor Horváth, Roland Dócs: Contributed reagents, materials, analysis tools or data.

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Data availability statement

Data included in article/supp. material/referenced in article.

Declaration of interest's statement

The authors declare no competing interests.

Nomenclature

mm	Millimeter
μm	Micrometer
N/mm^2	Newtons Per Square Millimeter
g/mm^2	Gram Per Square Millimeter
g/mm^3	Gram Per Cubic Millimeter
g/cm^3	Gram Per Cubic Centimeter
rpm	Revolutions Per Minute
g	Gram
m	Milliliter
lb/gal	Pound Per Gallon
1/s	Per Second
π	Pi is approximately (3.14)
KN/min	Kilonewton Per Minute
KN	Kilonewton
N	Newton
MPa	Mega Pascal
mD	Milli Darcy
cm	Centimeter
%	Per Cent
°C	Celsius

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