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Effect of saponification-based treatment of palm kernel shell aggregates on the mechanical properties of palm kernel shell aggregates concrete

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Highlights

- The induced saponification removed oil on the surface of PKS aggregates.
- Oil on PKS aggregates does not allow the development of strength.
- High alkalinity destroys the integrity of PKS aggregates.
- High alkalinity affect adversely mechanical strengths of PKS concrete.

Abstract

This paper presents a method of treatment of Palm Kernel Shell (PKS) aggregates to remove the oil that inhibits the development of strength in PKS concrete. 13 treatments from 2 to 20 g/L concentration of solutions of potassium hydroxide were used to induce a saponification reaction with the oil present on the PKS surface. The oil is entirely removable, but the highly concentrated solution damages the aggregates' surface. The optimum concentration to remove the oil was 10 g/L for an optimum exposure time of 12 h without affecting the PKS aggregates' integrity and the PKS concrete's mechanical properties. Compressive strength and a splitting tensile strength at 28 days of 20 MPa and 1.2 MPa were achieved without admixture.

Previous

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Keywords

Saponification; Palm kernel shell; Compressive strength; Splitting tensile strength; Palm Kernel Shell Concrete

1. Introduction

A United Nation (U.N). Environment report places the energy consumed by the construction sector at 36 %, which makes it the largest consumer of global energy, and 39 % is associated with CO_2 emissions [1]. Concrete occupies the first place among the building materials because of its history, which goes back thousands of years. Its versatility in use, resistance to environmental attack and ease of application, including as reinforced concrete, gives it many advantages of for use in infrastructure. Unfortunately, it is also the product that releases the most CO_2 in construction. Concrete alone accounts 8 % of global CO_2 emission [2]. One of the solutions to reduce the adverse environmental effects of the construction sector is to improve the efficiency of construction by using lighter construction material.

Concrete is made mainly with aggregates of mineral origin, representing 68 to 85 % of the volume of normal concrete [3], [4]. Materials extraction is estimated to be between 47 and 59 billion tonnes per year [5], and it is projected that by 2050, this rate would reach 258 billion tonnes [6]. Since these materials are non-renewable, finding an alternative is urgent. Apart from the big problem of sustainability, the extraction of materials has many other impacts, including biodiversity destruction, land losses, ground water supply destruction, among others. Part of the solutions to this issue is to replace mineral aggregates by industrial by-products such as slag from steel industry, recycled aggregates from demolition works [7], and agricultural waste like coconut shells, palm kernel shell, sawdust, cork [8], [9]. Palm kernel shells or coconut shells which are pretty common in sub-Saharan Africa, have been studied and have shown great potential to be used as aggregate in structural concrete.

Oil palm industry wastes are of great interest because this industry is growing yearly. Palm oil production moved from 56.38 million to 75.5 million metric tonnes worldwide from 2012 to 2022 [10], [11]. In 2018, more than 2.5 million tonnes of palm crops were produced in Africa, representing about 800,000 tonnes of palm kernel shells (PKS) [11]. PKS has been used as coarse aggregates for structural concrete and has shown great results [12], [13], [14], [15], [16],

[17], [18], [19]. Among the results, a 28-day compressive strength of 54.4 MPa, which shows that this aggregate can be used to produce high-strength concrete [17]. Better shear resistance compared to normal concrete was also observed [13], [14]. However, many researchers observed that this type of aggregate needs to be pre-treated to remove the oil on its surface. This will improve the bond between the aggregates and the paste [20], [21]. Therefore for the PKS aggregates, they have proposed many different methods of treatment, among them: coating to reduce the porosity [22], treatment with 5 % NaOH [23], and heat treatment on old PKS aggregates [24].

In this study, the process of saponification used for the production of traditional soap in Africa is used to treat the PKS aggregates for massive production, and the influence of this treatment on the properties of both PKS aggregate and PKS concrete was studied.

2. Materials and methods

2.1. Materials

The cement used for this study was CEMI 42.5 N produced in Kenya and satisfying the requirement of BS EN 197-1 [25].

The fine aggregates was river sand from Meru, Kenya. The sand was well graded as shown Fig. 1, and its characteristics summarized in Table 1 shows it was suitable to be used for the production of concrete of good quality.

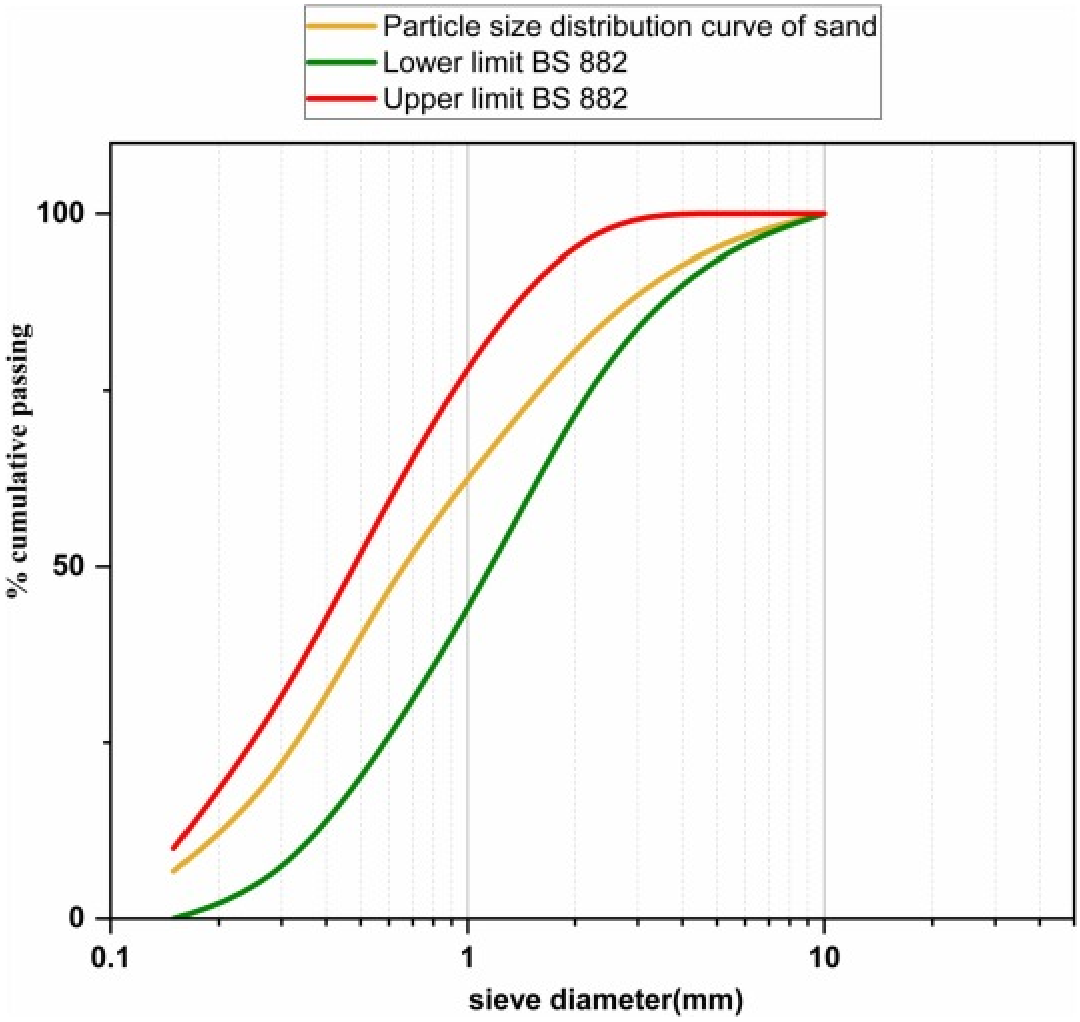

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Fig. 1. Particles size distribution of sand.

Table 1. Summary of the characteristics of fine and coarse aggregates.

Characteristics	Fine aggregates(sand)	Coarse aggregates (PKS)
Maximum aggregate size	5 mm	10 mm
Fineness modulus	2.76	–
Specific gravity	2.63	1.15
Saturated surface dry (SSD) density	2.72	1.32
Apparent density	2.86	1.43
Loose bulk density	1650 kg/m^3	560 kg/m^3

Characteristics	Fine aggregates(sand)	Coarse aggregates (PKS)
Compacted bulk density	1800 kg/m^3	644 kg/m^3
24 h Water absorption	2.75 %	25.7 %
Percentage of void (uncompacted basis)	–	47 %
Percentage of void (compacted basis)	–	38 %
Aggregate Impact Value (AIV)	–	5 %
Aggregate Crushing Value (ACV)	–	3 %
Flakiness Index	–	36

The palm kernel shell aggregates used as coarse aggregates were the wastes of palm oil processing and were collected in Uganda, at the border with the Democratic Republic of Congo. After the pulp was removed to produce the red oil as a primer product, the shells were manually or mechanically broken to remove the kernel or endosperm used to make palm kernel oil. The PKS collected had a maximum size of 20 mm. They were crushed with a plant milling machine to obtain aggregates of grade 5/10. The specie of kernel used for this research is the variety called Dura, which has a thick shell of 2 to 5 mm [17].

The particle size distribution analysis done according to BS EN 12,620 showed that the PKS was well graded and suitable for use as coarse aggregates based on the results obtained (Fig. 2). The PKS characteristics are summarized in Table 1. The values obtained in this study for specific gravity, apparent density, loose bulk density, 24 h water absorption, aggregate impact value (AIV) and aggregate crushing value (ACV) are in the range of the values reported in the literature [21], [26], [27], [28], [29] (Fig. 3).

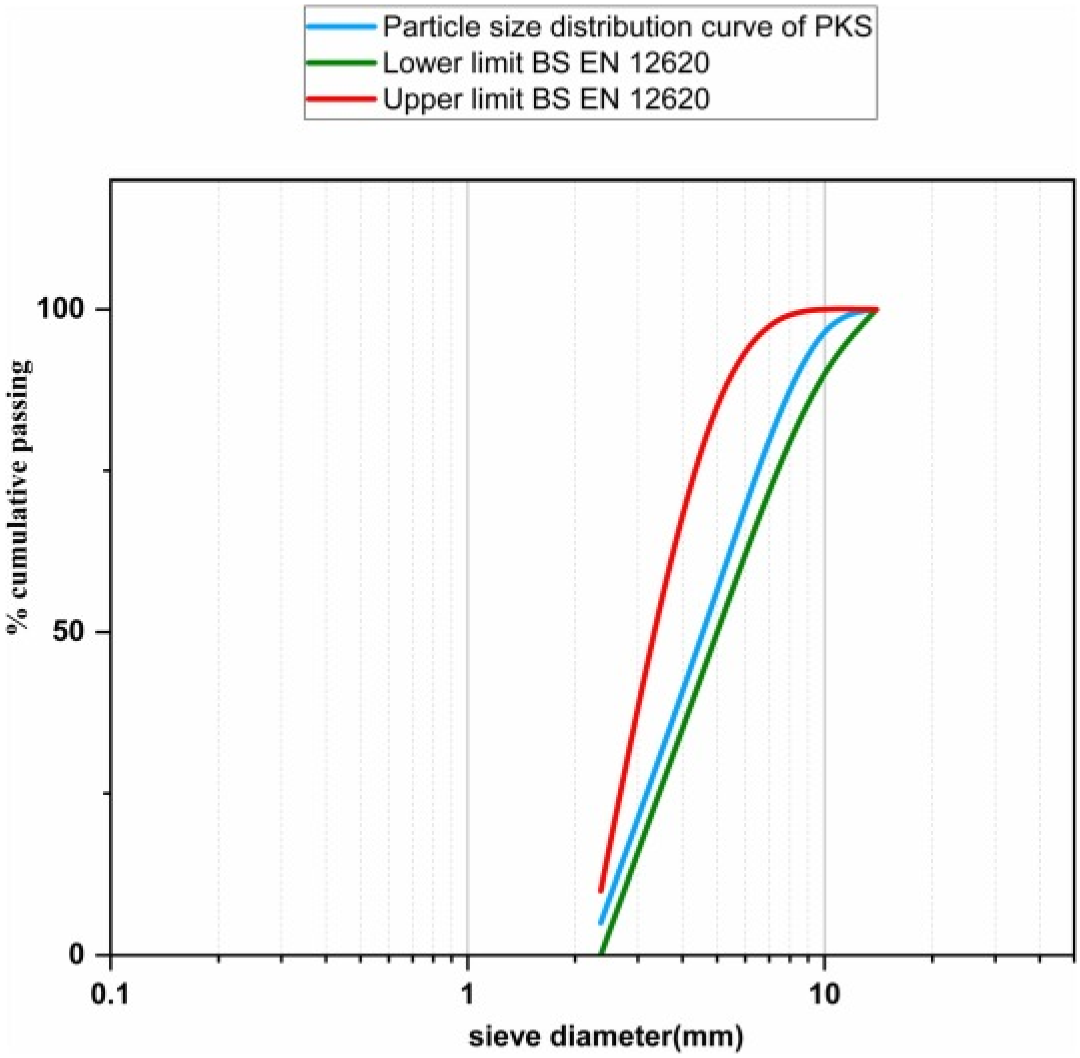

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Fig. 2. Particle size distribution of PKS.

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Fig. 3. PKS aggregates before and after treatment.

2.2. Saponification-based treatment of PKS aggregates

Naturally, the PKS have oils on their inner and external surfaces, and the process used aims to remove the oil using an induced process of saponification following equation (1) [30], [31], [32], which is called direct neutral fat saponification [33]. The oil on the surface of the PKS is due to triglyceride fat. These oils react with a strong base or alkali to produce glycerol and soap. This study used 85 % potassium hydroxide(KOH) as alkali at various concentrations and duration. The soap is generally extracted by precipitation in the presence of salt. For this study, both products glycerol and soap were simply washed out.

$$\begin{array}{lll} C\ H_2OR & & C\ H_2OH \\ \vert & & \vert \\ C\ \ HOR & +3KOH \rightarrow & C\ \ HOH\ \ +3RCOO^{-}K^{+} \\ \vert & & \vert \\ C\ H_2OR & & C\ H_2OH \\ \quad Fat & \quad Alkali & Glycerol \quad Soap \end{array} \tag{1}$$

The PKS collected were washed to remove the free fibres, the dust, and any substance of unknown origin prior to the saponification treatment. A few of the PKS aggregates still had the mesocarp fibres and a part of their kernels on the surface, even after this first wash. They were put saturated surface dry (SSD) state (Fig. 3a) before being soaked in different concentrations of potassium hydroxide solutions for 2 and 24 h. 13 treatments were conducted, as summarized in Table 2. After each treatment, the PKS aggregates were washed to remove the soap on the surface (Fig. 3b). During the saponification process, the water changed from a neutral colour to a dark colour. The soap was believed to be removed when the water used for washing remained clear. Shows the PKS before and after treatment.

Table 2. Concentration of KOH per treatment of PKS aggregates.

Label	T-0	T-2	T-3	T-4	T-5	T-6	T-7	T-8	T-10	T-12	T-14	T-16	T-18	T-20
Concentration (g/L)	0	2.4	3.4	4.4	5.4	6.4	7.4	8.4	10.4	12.4	14.4	16.4	18.4	20.4

2.3. Rate of absorption of PKS aggregates

After each treatment, the sample was sundried to a constant mass. The constant mass was achieved when the dry mass remained within 0.1 %. 200 g of the sundried sample W1 was immersed in distilled water for different time durations (15 min, 30 min, 60 min, 120 min, 360 min, 480 min, 600 min, 720 min and 1440 min). After the time elapsed, the sample was removed from the water, put at the SSD state and the weight W2 was taken. The quantity of absorbed water (H) was calculated following equation (2). The results gave a piece of information on the effect of the treatment on the absorption capacity of the PKS aggregate.

$$H = W2 - W1 \quad (2)$$

H is the quantity of absorbed water,

W2 is the SSD weight of the sample after immersion.

W1 is the weight of the sundried sample before immersion.

2.4. Crude fat content by Soxhlet extraction

The Soxhlet method for fat extraction is a traditionally used to extract fat using a solvent, especially in the food industry [34], [35]. To measure the effectiveness of the treatment on the reduction of the oil, the crude fat extraction by Soxhlet was carried out on 8(0, 8.4, 10.4, 12.4, 14.4, 16.4, 18.4, 20.4 g/L) of the treatments cited in Table 2. After each treatment, a dried sample of PKS was taken, crushed, sieved with a 300 µm sieve, and the particles passing were kept. 5 g(Ws) of each sample was placed in a thimble and refluxed for 8 h with the Soxhlet using 300 ml of 85 % hexane typeA.R.by Loba Chemie as solvent per sample. Each empty flask (W1) was weighed before adding the solvent. After the flask cooled, the hexane was removed using a rotary evaporator. The flask was oven dried for 30 min at 70 ± 5 °C, and cooled to ensure that only the fat/oil remained in the flask. The weight (W2) was then taken. The percentage of oil/fat extracted was calculated using fat % $=\frac{\mathrm{W2-W1}}{\mathrm{Ws}}\times 100$ Equation (3).

$$\mathrm{fatSom}\% = \frac{W2-W1}{Ws} \times 100 \quad (3)$$

W2 is the weight of flask + extracted oil,

W1 is the weight of empty flask.

Ws is the weight of the sample prior to test.

2.5. Scanning electron microscopy (SEM) of PKS aggregates

The Scanning electron microscopy (SEM) analysis was conducted on the same samples used to run the fat extraction by Soxhlet. This test aimed to visualize and understand the microscopy of the surface of the treated and untreated aggregates.

2.6. Properties of PKS concrete

The slump, compressive and split tensile strength were measured to determine the effect of the treatments on the physical and mechanical properties of the concrete. For each treatment the mix proportion used were C:S: PKS = 1:1.14:1.44 with w/c = 0.45.

The PKS used for the concrete mixing were put in a the SSD state to avoid the interference of the porous aggregates with the water used for concrete casting.

3. Results and discussion

3.1. Absorption of PKS aggregates vs treatment

Fig. 4 shows the absorption rate of the untreated (0 g/L) and treated PKS aggregates with different concentrations of potassium hydroxide (KOH) from 2.4 to 8.4 g/L. The curves from 2.4 to 7.4 g/L follow the shape of the control curve (untreated aggregates). The curves are continuous with no peaks or apparent slope change, meaning the absorption was not interrupted during the 24 h of absorption. The absorption of the control continued till 6 h where the curve flattened because there were no available pores to continue to absorb the water. All the treatments increased the rate of absorption of the PKS, but it was more predominant after 4.4 g/L (Fig. 4-a). At contact with water, the available pores of the aggregates immediately absorbed the water, which was observable with fast absorption during the first 80 min (Fig. 4-b). For 8.4 g/L, the absorption continued and presented a peak at 6 h of absorption, then dropped and afterwards to increase slowly after 12 h. This observation means that all the treatments below 8.4 g/L do not significantly affect the absorption, and consequently, the reduction of the oil content. More pores containing oil needed to be emptied to allow the absorption to continue. The observable peak at 6 h of absorption for a treatment at 8.4 g/L led to the study of the rate of absorption of treated PKS above 8.4 g/L in Fig. 5.

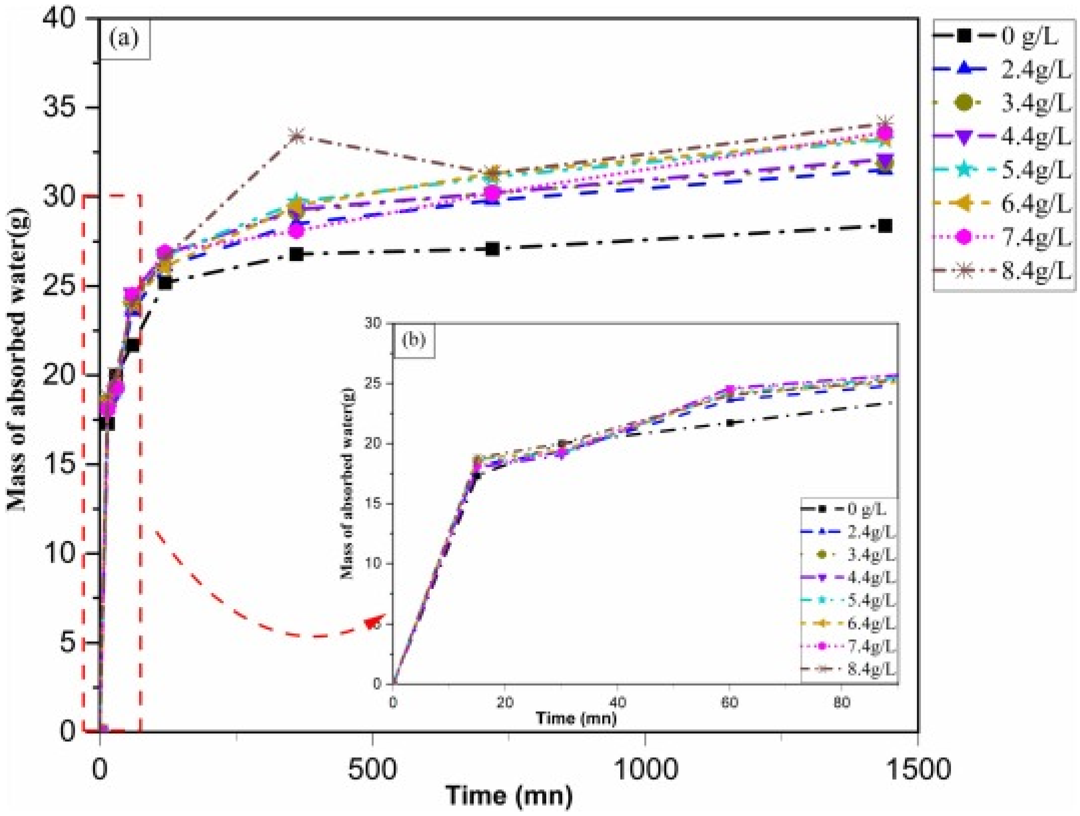

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Fig. 4. Absorption rate of water vs KOH treatment of PKS treated for 0, 2.4, 3.4, 4.4, 5.4, 6.4, 7.4 and 8.4 g/l of KOH under 24 h (a) and 2 h (b).

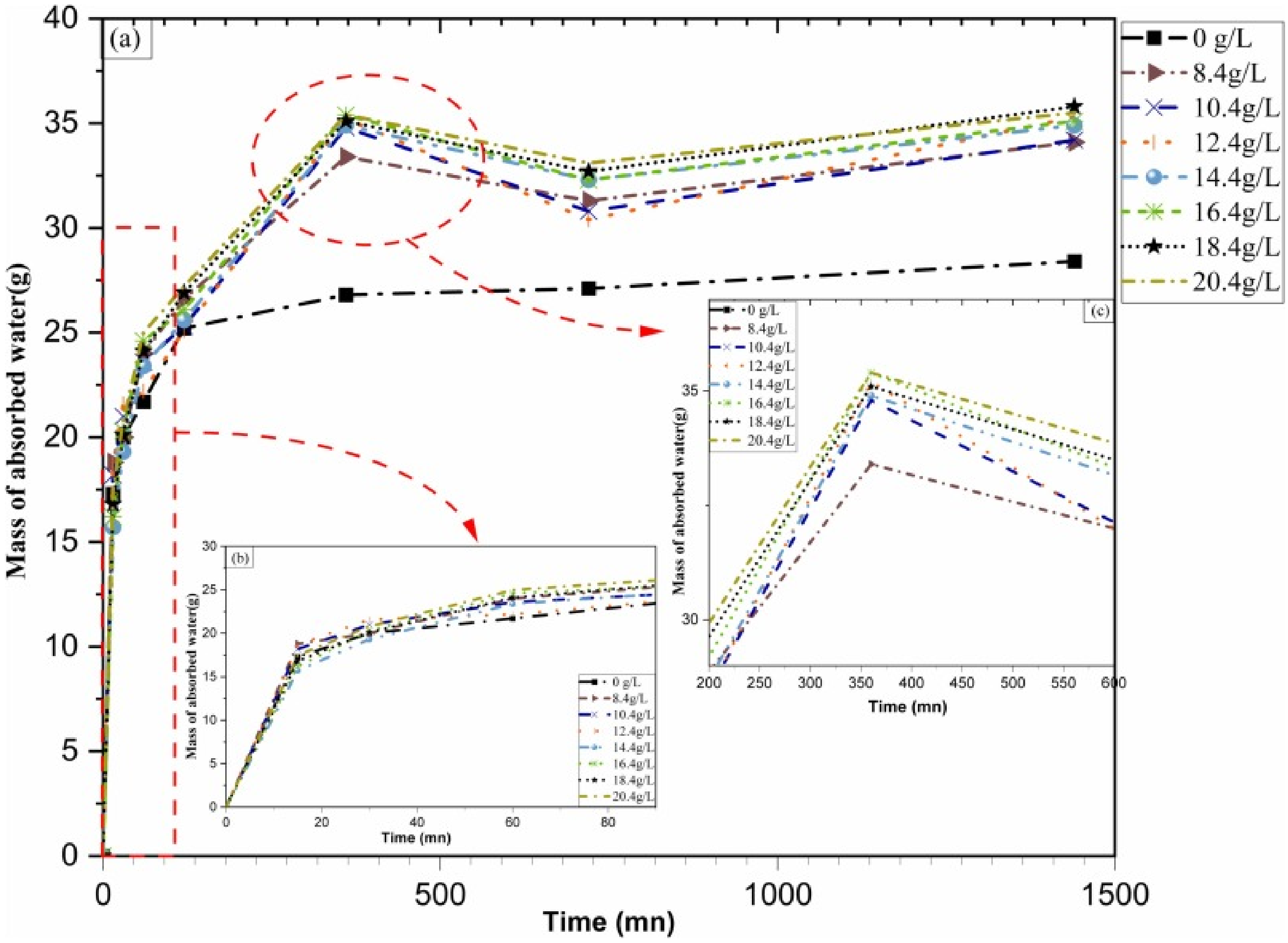

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Fig. 5. Absorption rate of water vs KOH treatment of PKS treated for 0, 8.4, 10.4, 12.4, 14.4, 16.4, 18.4, and 20.4 g/l of KOH under 24 h (a), 12 h (b), and 1 h(c).

The tests showed fast absorption during the first 80 min, as observed in Fig. 5 and previously in Fig. 4. 56 to 62 per cent of the water was consumed during the first 30 min, and about 75–85 % in the first hour (Fig. 4-b and Fig. 5-b). This observation is similar to the behaviour reported in the literature [15], [29], [36]. The results also justify the need for presoaking the PKS aggregate and their use at the SSD state, so absorption does not interfere with the process of hydration of cement that needs water. All the curves from 8.4 g/L showed a peak at 6 h followed by a weight loss till 12 h. After that, the absorption continued to increase for 24 h.

It will be recollected that the oils the treatment was meant to remove are from different origins. The inner part of the aggregates has on its surface the oil from the kernel that contributes to the kernel sticking on the surface. On the other hand, the outer part has the oil from the mesocarp. Sometimes, the mechanical process of extracting the kernel leaves a certain percentage of the kernel stuck on the inner part. It is also believed that after the treatment, a certain quantity of potassium hydroxide remains on the surface of the PKS. Therefore, during the absorption test, when in contact with water, the saponification reaction restarted and consumed the remaining oil, mainly from the stuck kernel. The kernel is composed of more than 80 % of fat [37], [38], which means it will lose about 80 per cent of its weight during the saponification reaction. The kernel's weight is reduced as the oil is consumed, which explains the PKS sample weight loss between 6 and 12 h. The saponification process is a slow reaction. Up to 6 h, the observed absorbed water is mainly due to the opened pores being free of oil after the first saponification reaction. This peak is nonexistent from 0 to 7.4 g/L because the concentration was insufficient to consume all the sources of oil. When the same test is conducted on samples of selected PKS without kernel on their surface, the curves do not show the peak at 6 h, as shown in Fig. 6. Since the saponification process is prolonged, this weight loss is progressive, which explains the 6 h whereby the weight was reduced. After 12 h, the maximum of the oil was consumed, the mass of the sample was stable, and the oil was removed from the pores allowing the absorption to continue.

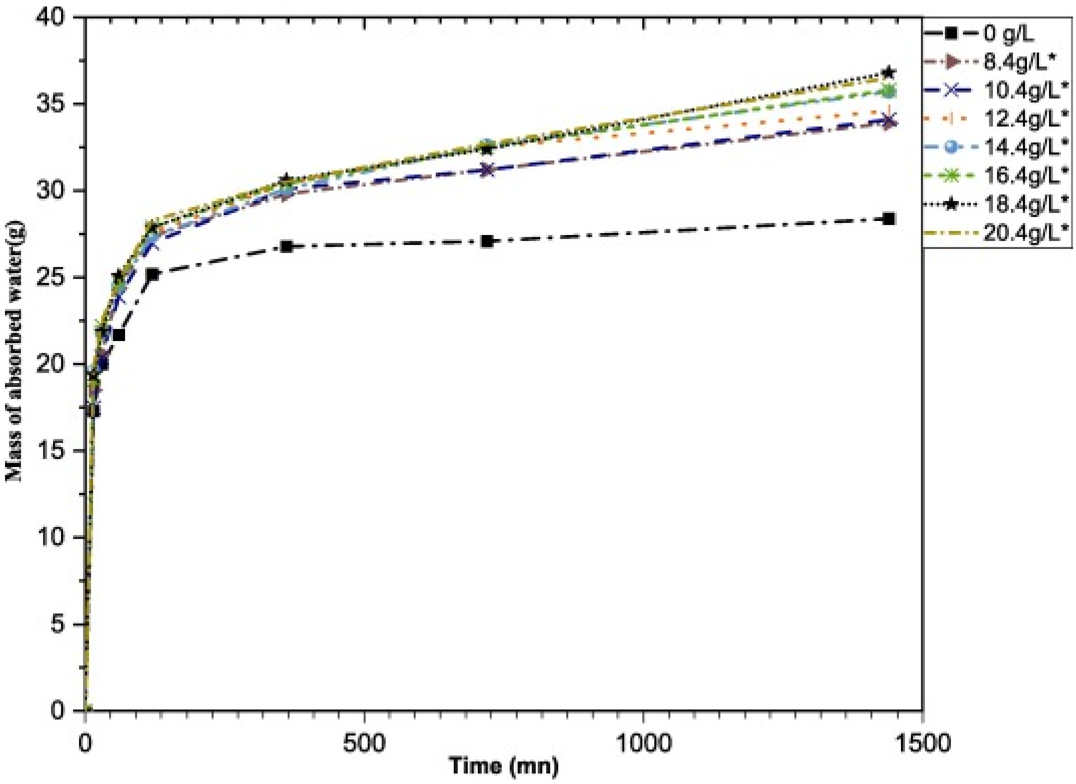

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Fig. 6. Rate of absorption of water vs KOH treatment of PKS without kernel treated for 0, 8.4, 10.4, 12.4, 14.4, 16.4, 18.4, and 20.4 g/l of KOH for 24 h.

The peak at 6 h for all the treatments is essential and might establish the point where the saponification process impacts the consumption of the different oils in the aggregates. Therefore, six hours is the minimum and 12 h the optimum time needed to consume the maximum of oil through the saponification process once the proper concentration is defined.

In Fig. 6, the PKS without kernel generally absorbed more water than the raw PKS with some kernels, which means that more pores are available in the absorption process. From 14.4 g/L to 20.4 g/L, the absorption rate is high, whether with or without kernel, compared to the other treatments, which led to the study of the microstructure in 3.2.

3.2. Microstructure of the PKS aggregates

The microstructure of the PKS aggregates (Fig. 7) shows two distinct parts: the kernel's surface (2) and the embedded fibers (1) with their extensive network of pores.

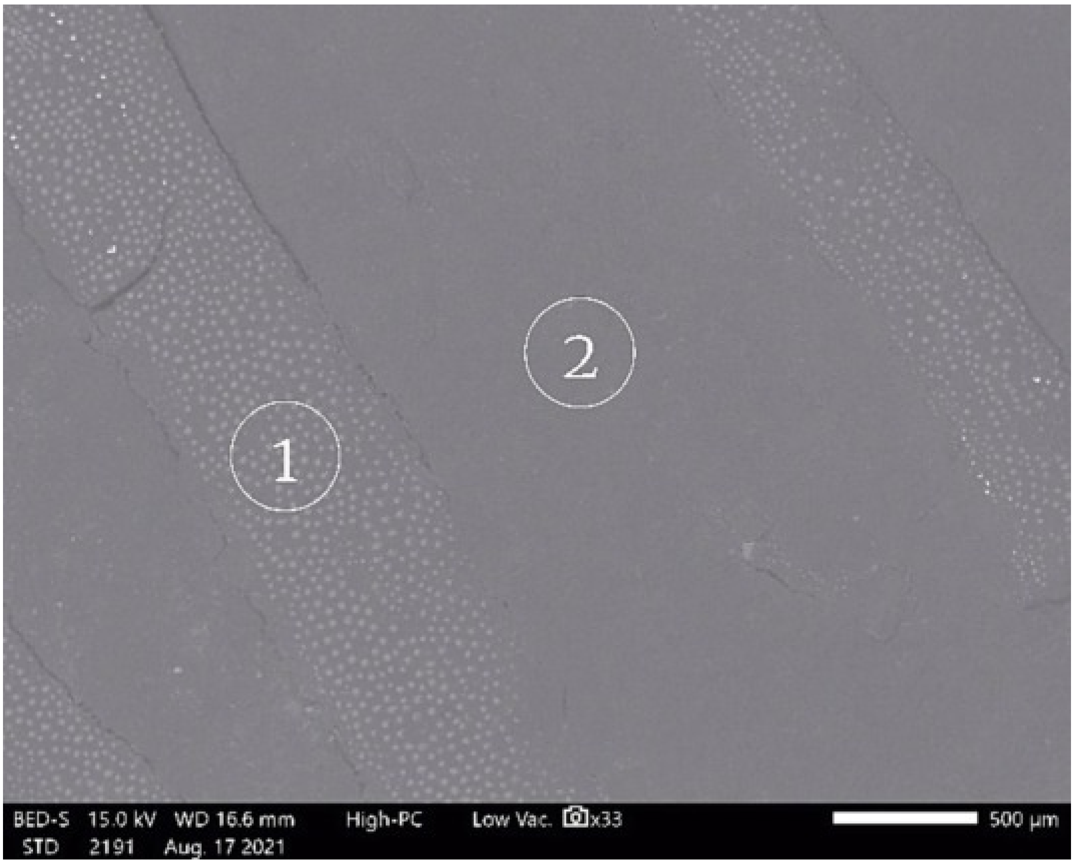

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Fig. 7. SEM on the surface of the untreated PKS aggregate at x33 magnification.

The embedded fibres have a large network of pores about 20 μm in diameter (Fig. 8-a). Both the pores and the surface of the fibers have oil. The shell of the PKS's aggregates is like an assembly of plates. It also has tiny pores compared to the pores of the embedded fibres (Fig. 8-b). When the oil occupies the inner part of the pores of the embedded fibers, the oil covers the pores and surface of the shells. The oil does not allow the pores to absorb the water because they cause an obstruction.

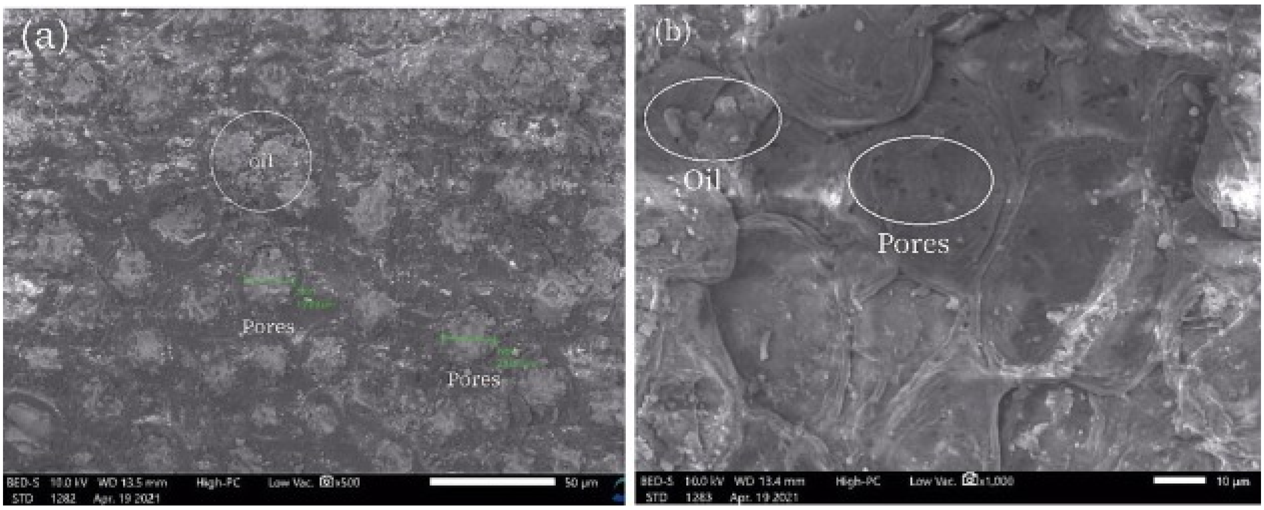

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Fig. 8. SEM analysis on the untreated PKS's embedded fibre at x500 magnification (a) and on the shell's surface at x1000 magnification(b).

Fig. 9 shows the surface of the PKS aggregate after treatment at 10.4 g/L. Compared to Fig. 8, the oils in the pores of the fibers (Fig. 9-a) have reduced and are almost inexistant on the surface of the shell(Fig. 9-b).

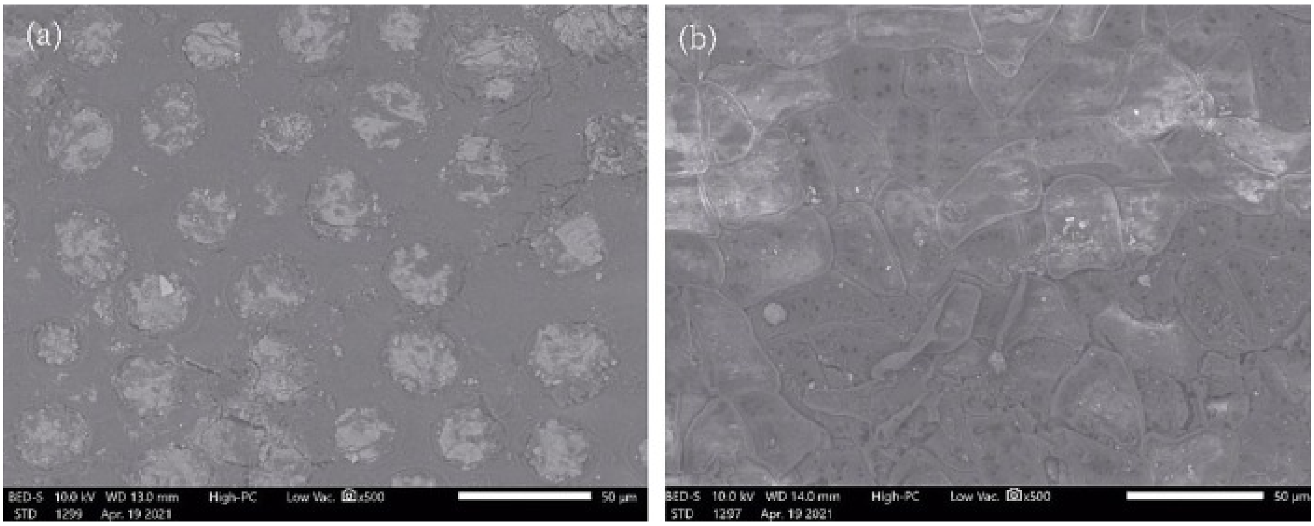

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Fig. 9. SEM analysis on the treated PKS with 10.4 g/L of KOH, PKS's embedded fiber surface at x500 magnification (a) and shell's surface at x500 magnification (b).

On the PKS treated at 20.4 g/L of KOH, the pores of the embedded fibres are empty of oil (Fig. 10-a). Degradations are visible on the surface of the shell (Fig. 10-b,c).

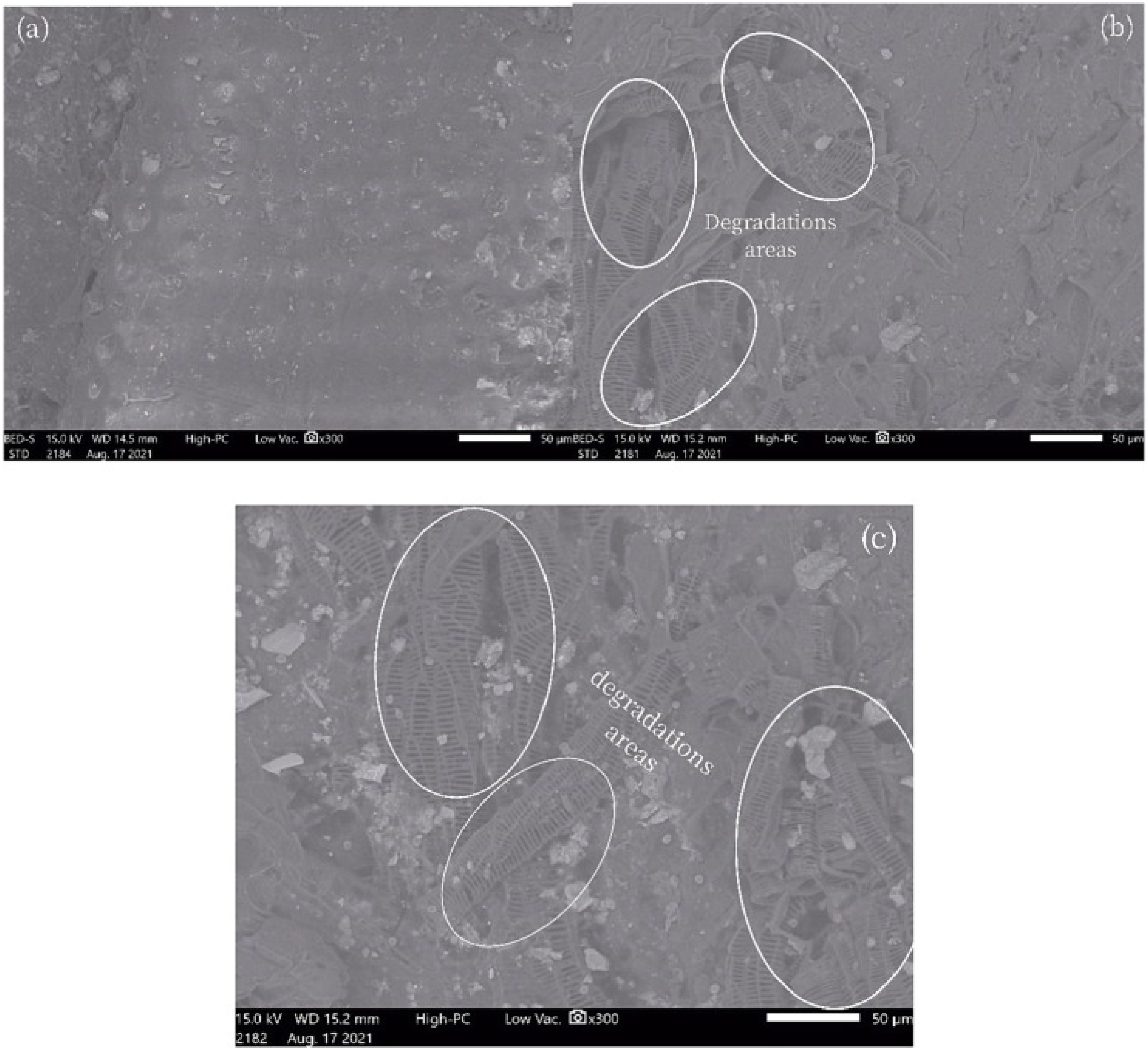

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Fig. 10. SEM analysis on the treated PKS with 20.4 g/L of KOH, PKS's embedded fibre surface at x300 magnification (a) and shell's surface at x300 magnification (b) L, and shell's surface at x300 magnification (b) at 18.4 g/L.

The microstructure analysis shows that the oil is reduced both on the shell's surface and inside the embedded fibres' pores. It can be concluded that the saponification process removed the oil present on the PKS.

Those degradations are already visible at 14.4 g/L treatment. The degradations visible on the surface of the shell (Fig. 10-b) showed that excessive use of KOH will destroy the shell's structure, affecting the quality of the PKS aggregates and, consequently, negatively affecting the performance of the concrete. It is believed that the white particles near the degradation zone are the remaining potassium hydroxide that was not removed after the treatment. It also informs that PKS concrete is sensitive to an alkaline environment which may reduce its performance.

For further studies, adding some heat during the saponification process could be of interest to accelerate the process and avoid any remaining potassium hydroxide.

3.3. Crude fat extraction

The crude fat content was determined by Soxhlet method at 2 h of treatment as proposed in the literature [18], [39] and at 24 h treatment as part of this study and the results are plotted in Fig. 11. The crude fat extraction aimed to ensure that the oil in the pores and on the surface of the aggregates is removed (Fig. 12).

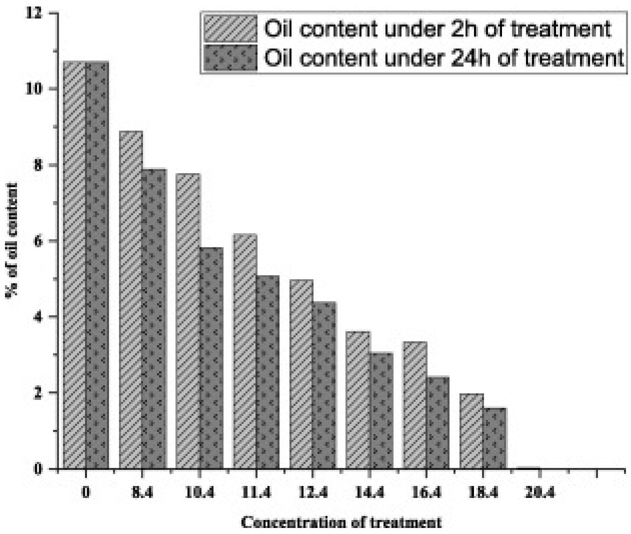

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Fig. 11. KOH treatment vs oil content.

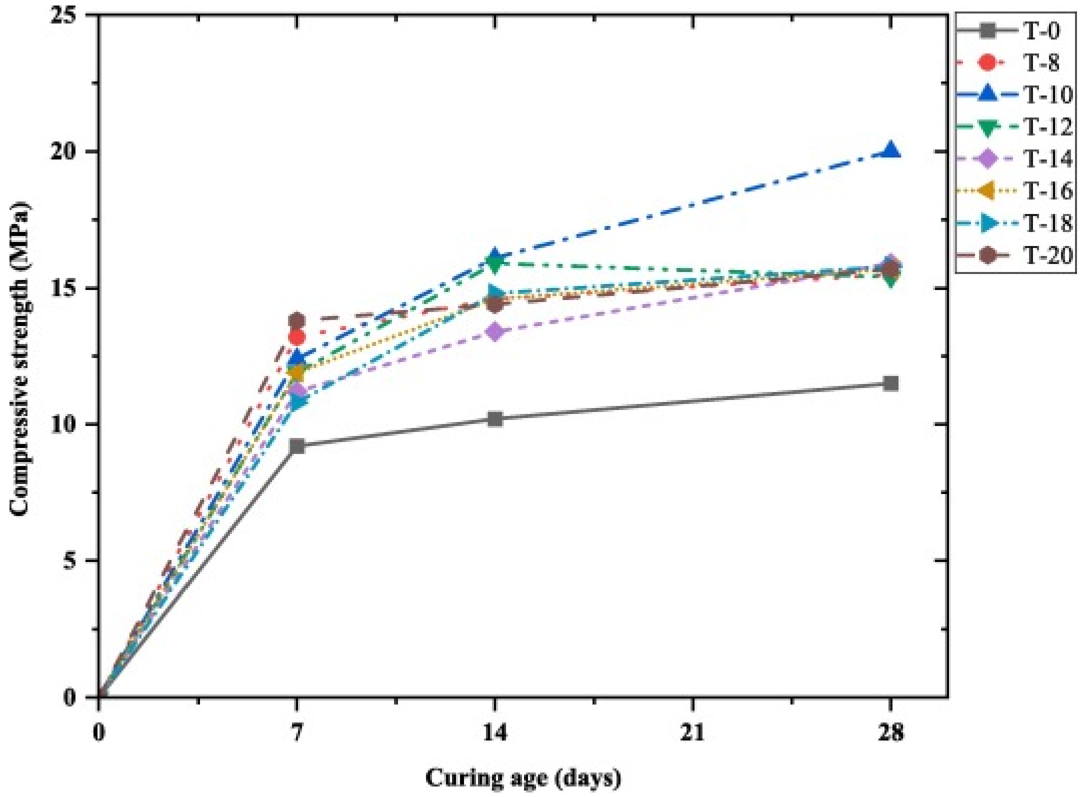

Fig. 12. Compressive strength vs curing age.

For all the treatments, the oil content was reduced with the increase of the concentration of potassium hydroxide. The treatment at 24 h has a better effect on reducing the fat (oil) content than the 2 h treatment proposed in the literature [39]. It is because saponification is a slow process. This slow reaction, coupled with the observation made on 3.1, requires a minimum of 6 h to reduce the minimum of oil (Fig 11).

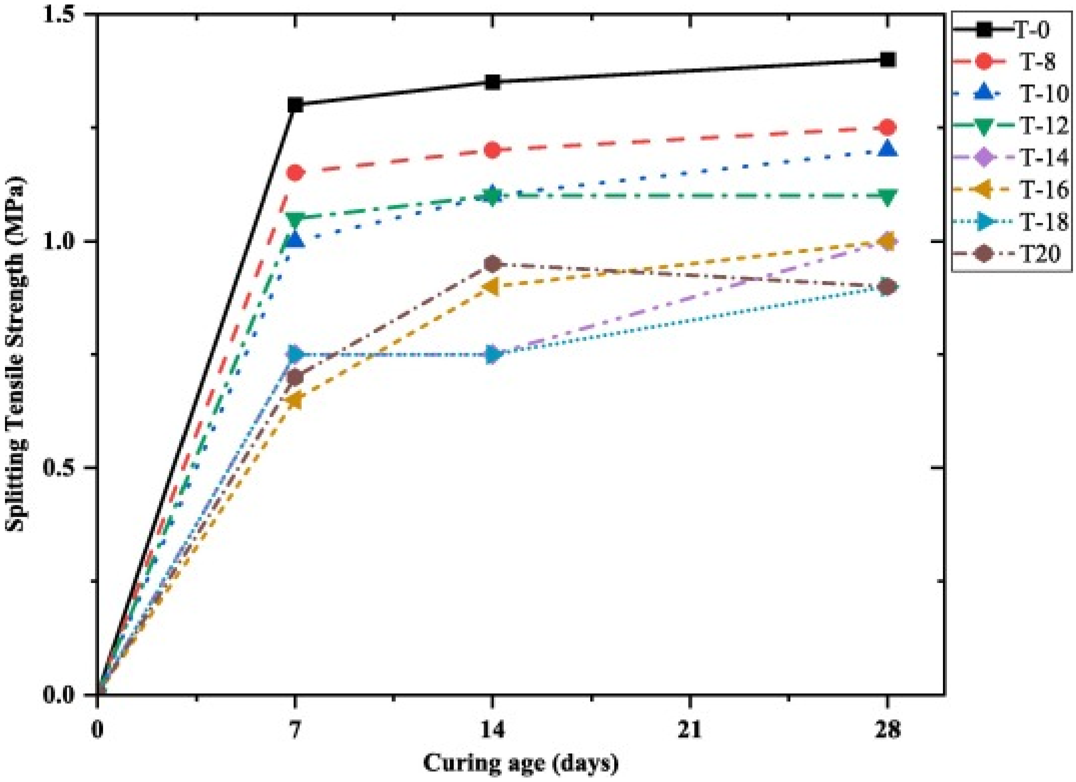

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Fig. 13. Splitting tensile strength vs curing age.

At 20.4 g/L, the crude oil extraction showed that the presence of oil was nil. Beyond this concentration, a treatment will create more damage on the aggregate surface. The removal of the oil is important so that it does not interfere with the process of hydration and the development of strength [40], [41].

3.4. Properties of PKS concrete

3.4.1. Slump

T-0, T-8, T-10, T-12, T-14, T-16, T-18, and T-20 have respectively values of slump of 72 mm, 73 mm, 75 mm, 100 mm, 143 mm, 89 mm, 84 mm and 60 mm. According to BS EN 206 [42], all are class S2 except T-12 and T-14, which belong to class S3. Nevertheless, all the concrete made satisfied the requirement of workability.

3.4.2. Compressive strength

The treatment of PKS aggregate has shown that the nontreatment of the PKS impacts the development of the strength. The oil interferes with the development causing low compressive strength values with the untreated PKS. The highest compressive strength value at 28 days was obtained at the treatment of 10.4 g/L. Although the compressive strength at 7 days is not the highest, the ratio fc7/fc28 = 0.62 is similar to the one reported in the literature [28], [43], [44], which means that the development of the strength is normal at treatment of 10.4 g/L concentration of potassium hydroxide. For all the other treatments, the curves almost flattened after 14 days, which shows that the strength development is disturbed. Below T-10, the quantity of oil removed is insufficient to ensure strength development. The degradation observed on the surface of the aggregates at T-14 caused the loss of strength development beyond T-14 (Fig. 12).

The splitting tensile strength of PKS aggregate concrete made in this study varied from 0.9 MPa to 1.4 MPa at 28 days (Fig. 13). The untreated aggregate gives the highest value due to free fibres on the surface. As the free fibres fixed on the surface of the PKS by the oil are removed after the saponification process, the splitting tensile strength is reduced, as seen in the results of the splitting tensile strength at 7 days. In the literature, the splitting tensile strength represents 6 to 10 % of the compressive strength [16], [29]. Based on this observation, the treatment at 18, 20, and the untreated aggregates are not satisfying the behaviour of PKS aggregates concrete reported in the literature. T-8 and T-12 show similar behaviour to the untreated aggregates. The early strength is high and has a little progression after 7 days. T-14 and T18 also have the same behaviour. There is no change in the strength between 7 and 14 days followed by a significant increase after 14 days. The results after T-14 are unreliable because the aggregates already have some surface degradations due to the treatment. This might be the reason for the inconsistent behaviour starting from this value. This drop in the strength of T-20 can be be explained by the degradations that were more predominant in this sample than any other treatment. The rise in strength in T-16 and T-20 between 7 and 14 days is not expected and require further investigation.

4. Conclusion

- The saponification-based treatment effectively reduced the oil content on the surface of the PKS aggregates.

- The reduction of oil is effective after a minimum treatment of 6 h and an optimum of 12 h. These values might be improved with heat applied during the reaction.

- The washing process has to be improved to remove the entire potassium hydroxide that can affect the integrity of the PKS.

- The study established that the optimum KOH concentration for treatment is 10.4 g/L. Beyond this value, the PKS aggregates are damaged by the alkaline attack, which creates a loss of compressive strength and inconsistency in the splitting tensile strength.

CRediT authorship contribution statement

Ahouefa Reine Katte: Conceptualization, Methodology, Resources, Investigation, Writing – original draft, Funding acquisition. **John Mwero:** Validation, Supervision, Writing – review & editing. **Mohamed Gibigaye:** Validation, Supervision, Writing – review & editing. **David Otieno Koteng:** Validation, Supervision.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Ahouefa Reine KATTE reports financial support, administrative support, and equipment, drugs, or supplies were provided by Pan African University. Ahouefa Reine KATTE reports equipment, drugs, or supplies was provided by Laboratory of food fortification.

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The microstructure analysis and crude fat tests were carried out at the Laboratory of food fortification-Jomo Kenyatta University of Agriculture and Technology (JKUAT) – Kenya.

Recommended articles

Data availability

Data will be made available on request.

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