An investigation of the effect of walnut shell as sand replacement on the performance of cement mortar subjected to elevated temperatures

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Abstract. This study examined the effect of ground shells of walnut (GW) as fine aggregate on the fresh and hardened properties of cement mortar before and after being subjected to elevated temperatures. The experimental work consists of two series with different water to cement ratio (w/c) and various percentages of GW. In each series, the ratios of GW were varied in range (0-30% at an increment of 10%). The fresh density and slump test were used as fresh properties and the dry density with compressive strength were measured at the curing ages of 7, 14 and 28 days as hardened characteristics. Also, the dry density and compressive strength at 28 days curing age were examined after exposure to an elevated temperature of 400 °C and 600 °C for two hours. The results indicated that all tested properties were reduced by using GW. The optimum utilized ratio of GW is 20% for the first series with w/c of 0.5 which led to producing lightweight cement mortar and is suitable for structural purposes before and after exposure to 400 °C. However, the rest of the mixtures are suitable for non-structural purposes.

1. Introduction
Walnut consumes in preparing a food supplement and the sweets in all cities of Iraq. The resulted waste of walnut’s shell can be around 43%-65% of the total weight of walnut depending on its size; which requires considerable land for its disposal. Utilizing these walnut’s shell wastes in manufacturing industrial materials like constructing material can be a safe solution for removing them. Where, the researchers had been investigated the consequence of the usage of different agricultures wastes in cement mortar and concrete.

The Agrarian harsh environments which can be utilized in mortar or concrete as a substitution of sand are groundnut shell (GNS), oyster shell (OS), sugarcane bagasse ash (SBA), giant reed ash (GRA), tobacco waste (TW), rice husk ash (RHA), sawdust (SD) and cork (C). These harsh environments require different methods for change them into sand. Some of them have pozzolanic
activity when it utilized in mortar or concrete; like RHA, GRA and SBA [1]. Alsalami [2] studied the absorption, density and compressive strength of cement mortar made by the waste of pistachio’s shells as fractional substitution of fine aggregate. She found the density and compressive strength decreased via using shells and the minimum density was 1000 kg/m³ at a 60% ratio.

Gil-Lim et al. [3] conducted investigation on studying the properties of oyster shell (OS) and they found that OS is active substitution of fine aggregate and a source of unadulterated materials of calcareous materials. Yoon et al. [4] used oyster shell (OS) as a sand substitution and found there was no considerable change in compression strength when sand replaced by OS ratio reached 40%. Li et al. and Yang et al. reported that the maximum OS weight ratio substituted as sand is 20% [5, 6]. Besides, other researchers found that OS used as aggregates led to upsurge shrinkage and decrease workability [6-8]. But, Yang et al and Mo et al [6, 9] mentioned that strength of mortar or concrete contained OS as a substitution of fine aggregate reduced by increase weight ratio of OS from 5% to 20 %.

Teo et al [10] conducted a study on using the shell of oil palm (SOP) as gravel in concrete and they found the possibility of utilized this SOP if the required concrete strength equal or less than moderate value 28 MPa. Also, Oyejobi et al [11] found that SOP can be utilized as gravel in the production of structural concrete with strength of 20.1 MPa when the blend of concrete is 1-1.5-3. Muthusamy and Zamri [12] investigated using SOP in concrete and they found that 20% of SOP can give suitable compressive strength. Modani and Vyawahare [13] investigated using the ash of sugarcane bagasse (ASB) as a substitution of sand and they found there was no reduction in strength and workability of concrete. Also, they indicated that 10% and 20% of ASB can lead to improving compressive strength.

Lim et al. [14] conducted an experimental study on using SOP as a filler in concrete and they found there was an increase in strengths of tensile, flexure and compression if the ratio of SOP ranged from 10%-20% compared to concrete made through utilize 100% normal fine aggregate as a filler. Sor N [15] studied usage pumice aggregate in self-compacting lightweight concrete production and found the compressive strength were reduced with the upsurge of pumice ratio. Sada et al. [16] conducted a study on usage groundnut shell (GNS) as a substitution of the sand and their results indicated the density, compressive strength and workability decreased with the upsurge of GNS ratio. Öz et al. [17] investigated the usage of lightweight aggregate manufactured by fly ash as fine and coarse aggregates for the blending of self-compacting lightweight concrete and the results indicated the reduction of density and mechanical strengths when the ratio of manufactured lightweight aggregate was upsurge. Mermersda et al. [18] studied the effect of cold-bonded fly ash aggregate on dry density and mechanical properties of geopolymer mortar. They found that the dry density and mechanical strengths reduced by increasing amount of manufactured fine aggregate in the mixtures. Hilal et al. [19] investigated usage of expanded polystyrene beads as a substitution of coarse in self-compacting concrete (SCC). They found the increasing usage of expanded polystyrene beads led to decrease the density, mechanical properties and velocity of ultra-sonic.

Mageswari and Vidivelli [20] investigated using SD ash (SDA) as a replacement of the sand in production of concrete. They stated that SDA can be suitable replacement of the sand and useful in decreasing the problems of dumping at concrete. Hilal et al. [21] utilized walnut shell (WS) as a substitution of gravel in production of SCC. Their findings indicated that using WS led to reduce the investigated mechanical properties of SCC. They recommended that (35- 50) % of WS can be used in produce light-weight SCC.

Hilal et al. [22] conducted a study on using WS in concrete as a replacement of gravel and sand. Their study consisted of three groups; at first one they substituted sand by WS in previous ratios; at second one they substituted gravel by WS in earlier ratios and they substituted sand and gravel by WS in the same previous ratios. They tested dry density, absorption ratio, splitting, flexural and compressive strengths. They found that the best properties were attained for the third group, and the best ratio of WS is 15% for this group.

The mortar has an important role in stresses distribution in the block building structure [23]. The fine aggregate has a significant influence on mortar’s proprieties as it is considered the main competent of the mortar and the selection of appropriate fine aggregate is so important at mortar’s
construction [24]. As Lenczner [25] mentioned, the mortar can give protection from water and air penetration when it uses as a bonding layer in blocks building. Besides that, the used mortar can recompense any variance in block dimensions and give the building some aesthetic impact. ASTM C 270 [26] classifies the mortar into four classes depending on its properties and its applications. The uppermost compressive strength mortar is Type M and low compressive strength is Type O.

The principal purpose of the current research is to evaluate the benefit of using ground walnut shell waste (GW) as a substitution of fine aggregate in mortar’s production. It predicted usage of GW can be given a light-weight mortar. The fresh density, slump and water absorption were found. Moreover, dry density and compressive strength at age of 7, 14 and 28 days were tested. The effect of elevated temperature on dry density and compressive strength was examined.

2. Experimental study and materials
2.1. Materials
2.1.1. Cement: Ordinary Portland Cement (OPC) type I that confirming to Iraqi specifications IQS No.5/1984 [27] was utilized this study. Physical and chemical properties presented in Table 1 and 2, respectively.

| Table 1. Physical properties of cement |
|---------------------------------------|
| Physical properties                  | Test result | Limits of Iraqi Specification No.5/1984 [26] |
| Setting time(minutes)                |             |                                             |
| Initial setting                      | 120         | 45 minutes                                 |
| Final setting                        | 360         | ≤ 600 minutes                               |
| Fineness by Blaine method (m² / kg)  | 300         | ≥ 230                                      |
| % Auto Clave                         | 0.31        | ≤ 0.8                                      |

| Table 2. Chemical compositions of cement |
|------------------------------------------|
| Oxide                                    | Weight (%) | Limits of Iraqi Specification No.5/1984 [26] |
| CaO                                      | 62.3       | -                                           |
| SiO₂                                     | 20.28      | -                                           |
| Al₂O₃                                    | 5.55       | -                                           |
| Fe₂O₃                                    | 4.20       | -                                           |
| MgO                                      | 2.60       | < 5.0                                       |
| K₂O                                      | 0.75       | -                                           |
| Na₂O                                     | 0.4        | -                                           |
| SO₃                                      | 2.5        | < 2.8                                       |
| Lime saturation factor                   | 0.81       | 0.66 – 1.02                                 |
| Insoluble Remains                       | 0.5        | < 1.5 %                                     |
| F.L                                      | 0.65       | -                                           |
| Total                                    | 99.63      | -                                           |
| Compound                                 | Weight (%) | Limits of Iraqi Specification No.5/1984     |
| C3S                                      | 50.05      | -                                           |
| C2S                                      | 20.45      | -                                           |
| C3A                                      | 4.05       | -                                           |
| C4AF                                     | 13.20      | -                                           |
2.1.2. Sand: The sand of a maximum size of 2.36 mm was used through this study, where it was transported from the AL-Habnya district. The physical properties and grading of utilized sand were followed to IQS No. 45/1984 [28] as can be seen in Table 3.

Table 3 Physical properties and grading of utilized sand

| Sieve Size | Passing% | IQs No.45/1984 |
|------------|----------|----------------|
| 2.36       | 100      | 100            |
| 1.18       | 87       | 89-100         |
| 0.6        | 65       | 60-100         |
| 0.3        | 20       | 30-100         |
| 0.15       | 5        | 15-100         |
| 0.075      | 0.5      | 5-70           |
| Pan        | 0        | 0-15           |

| Physical properties          |     |
|-------------------------------|-----|
| Specific gravity              | 2.65|
| Sulphate content (SO3%)       | 0.43%|
| Absorption%                   | 0.18%|
| Fineness modulus              | 3.05|

2.1.3. Walnut shells: The shells of walnut (SW) were collected from waste of consumed walnut at Anbar government. The particles of SW were cleaned by water, then left to dry by exposure to sun rays for at least one week. The dried SW’s particles were crushed through using mill apparatus to convert into smaller size that comparable to used sand as shown in Figure 1. These particles of ground walnut’s shells (GW) had been sieved and they had grading shown in table 4 which be less than the minimum requirement of [28]. Selected physical properties of GW are showed in Table 4. The chemical component of GW can be seen in Figure 2. The particle size distribution of the sand and ground walnut shells are seen in Figure 3.
Table 4. The Gradient of used walnut shells

| Sieve Size | Passing% | IQs No.45/1984 |
|------------|----------|----------------|
| 2.36       | 100      | 100            |
| 1.18       | 70       | 89-100         |
| 0.6        | 46       | 60-100         |
| 0.3        | 23       | 30-100         |
| 0.15       | 9        | 15-100         |
| 0.075      | 2        | 5-70           |
| Pan        | 0        | 0-15           |

Physical properties
- Specific gravity: 1.23 gm/cm³
- Absorption%: 0.21
- Fineness modulus: 2.5

Figure 2. The chemical component of GW, (a) The Spectrum of used GW in this study and (b) the electronic image.
2.1.4. Water: Tap water which is free from impurities was used for mixing and curing of all specimens at this study.

2.2. Mix design

The experimental program of this study was consisting of two series. Where, the proportion of mixing was 1:2.88 (cement: sand) for all mixes, and water to cement ratio was variable as shown in Table 5. For every series, the GW’s ratios were 10%, 20%, and 30% of the volume of sand. The symbol for each mix consists of one letter and two numbers; the letters R, B, and C refer to reference mix, the mix of the first series of 0.5 w/c with GW and mix of the second series of 0.4 w/c with GW. The first number indicates the series’ number and the second one refers to the ratio of GW.

Table 5. The Mix proportion in kg/m³

| Ratio of walnut % | Mix ID | Cement [kg] | Sand [kg] | Water [kg] | Walnut Shell [kg] | W/C |
|------------------|--------|-------------|-----------|------------|------------------|-----|
| 0                | R10    | 525         | 1512.7    | 262.5      | 0                | 0.5 |
| 10               | B11    | 525         | 1361.4    | 262.5      | 70.2             | 0.5 |
| 20               | B12    | 525         | 1210.2    | 262.5      | 140.4            | 0.5 |
| 30               | B13    | 525         | 1058.9    | 262.5      | 210.6            | 0.5 |
| 0                | R20    | 525         | 1512.7    | 210        | 0                | 0.4 |
| 10               | C21    | 525         | 1361.4    | 210        | 70.2             | 0.4 |
| 20               | C22    | 525         | 1210.2    | 210        | 140.4            | 0.4 |
| 30               | C23    | 525         | 1058.9    | 210        | 210.6            | 0.4 |

2.3. Testing procedures

2.3.1. Workability: The slump test was used to measure the mortar’s workability that conforms to instructions of ASTM C1437 – 15 [29].
Figure 4. Slump flow test.

2.3.2. Fresh and dry density: The density in the fresh and hardened state was measured according to the procedures of BS EN 1015-6 [30].

2.3.3. Compressive strength: The strength was tested via using cubes of 50 mm at ages 7 days, 14 days, and 28 days. The calculations were done by using three specimens for each mixes at all curing ages. the test conformed to rules of ASTM C109 [31]

2.3.4. Exposure to high temperature: After 28 days of curing, the specimens were put out from the curing tank and left to be dried for a day at room temperature. Then, the specimens were put in the oven for one hour at 100°C to assure is completely drying to prevent any explosion. Afterward, the specimens left to be cold at room temperature. Finally, the specimens were left in the oven for two hour after reaching the target temperatures which are 400 °C or 600 °C. The process of exposure to elevated temperature was done according to the procedure mentioned in [32].

3. Results and discussion

3.1. Fresh properties

Table 6 and Figures 5, 6 show the fresh properties of two tested series. The fresh density reduced by increasing the ratio of GW as a result of GW has a density lesser than utilized fine aggregate, and decreasing of w/c has an effect on the reduction of density. The slump values decreased sharply with the upsurge of GW ratio, and a decrease of w/c has a clear influence on this reduction which can be attributed to higher water absorption of GW compared with normal sand as shown in Figure 6. The maximum ratio of reduction of slump values was 98% for mixes B13 and C23. While the highest ratio of decreasing fresh density was 19% and 22% for mixes B13 and C23 respectively.

| Ratio of walnut% | Mix ID | Slump [cm] | Fresh density [kg/m³] |
|-----------------|--------|------------|-----------------------|
| 0               | R10    | 25         | 2300                  |
| 10              | B11    | 20         | 2056                  |
| 20              | B12    | 15         | 2008                  |
| 30              | B13    | *          | 1888                  |
| 0               | R20    | 22         | 2246                  |
| 10              | C21    | 10         | 2080                  |
| 20              | C22    | 12         | 1984                  |
| 30              | C23    | *          | 1808                  |
3.2. Hardening properties

3.2.1. Dry density

Table 7 and Figure 7 show the dry density of the two tested series. The dry density reduced with the increase in the ratio of substitutions of sand by GW. This result concurs with the findings by researchers [16, 17 and 21]. For the first series, the mortar with less and equal to 20% GW could be conformed to instructions of ASTM C270 [26] for loading bearing structures. For the second series, a ratio that less and equal to 10% of GW can attain these requirements of [26]. For two series, the ratio of GW more than 10% gave lightweight mortar according to the BS EN 998-2 [33]. Figures 8 and 9 show that density upsurge with age as a result of cement hydration and pores filling which makes the mortar has more weight [34]. The maximum rate of dry density development is 6% for R10 and the minimum one is 1.4% for R20. The density reduced with the upsurge of the GW ratio since the density of ground walnut shells is smaller than the density of normal fine aggregate. The highest ratio of decreasing dry density was 22% and 24% for mixes B13 and C23 respectively.
Table 7. Hardened properties of mortar at room temperature

| Ratio of walnut (%) | MIX ID | Dry Density (kg/m³) | Compressive strength (MPa) |
|---------------------|--------|---------------------|---------------------------|
|                     |        | 7 day | 14 da | 28 day | 7 day | 14 da | 28 day |
| 0                   | R10    | 2064  | 2102  | 2196   | 33    | 40.55 | 42.59 |
| 10                  | B11    | 1916  | 2004  | 1996   | 25.4  | 29.5  | 30.57 |
| 20                  | B12    | 1750  | 1784  | 1784   | 13.75 | 17.4  | 20.57 |
| 30                  | B13    | 1672  | 1720  | 1704   | 4.5   | 6.5   | 6.03  |
| 0                   | R20    | 2208  | 2238  | 2240   | 37.25 | 43.9  | 56.33 |
| 10                  | C21    | 1976  | 1996  | 2056   | 22.5  | 25    | 29.22 |
| 20                  | C22    | 1848  | 1888  | 1928   | 11.6  | 12.63 | 13.5  |
| 30                  | C23    | 1664  | 1676  | 1712   | 2.4   | 3.8   | 7.08  |

Figure 7. Relationship between dry density and GW ratios for two series.
3.2.2. Compressive strength

Table 7 and Figure 10 show the compressive strength values for two tested series. It is obvious that compressive strength reduced with the increased ratio of substitutions of sand by GW as a result of decreasing in workability besides the density and strength of GW are less than corresponding values of normal fine aggregate. The gotten result conformed to the findings of [16 – 19, and 21]. The reduction in compressive strength of the first series is less than this reduction of the second series which attributed to the low workability of the second series compared with the first series that led to produce a micro porous inside the structure of dry concrete contained GW.

The maximum ratio of reduction of compressive strength was 84% and 91% for mixes B13 and C23 respectively. The compressive strength for all mixes was developed with age in values different for each mix as a result of different cement hydration with age for each blend [34]. The increasing of GW ratio led to a decrease of compressive strength development with age because the reduction of workability and packing of the mix with the upsurge of GW ratio. The maximum rate of compressive strength development between the ages 28 days and 7 days is 66% for C23 and the minimum one is 14% for C22, see figures 11 and 12.
Figure 10. Relationship between compressive strength and GW ratios for two series.

Figure 11. Variation of compressive strength with the age for first series.
3.3. The properties after exposure to elevated temperature

3.3.1. Dry density

Tables 8 and 9 present the values and reduction of density after exposure to elevated temperature at 400 and 600 °C then graphically demonstrated in Figures 13 and 14, respectively. It can be observed that the reduction in density increased with an upsurge in the ratio of GW, and this reduction for the second series is slightly more compared with the first series. This upsurge in a reduction of density could be attributed to the existence of more voids as the ratio of GW increased with less weight.

**Table 8. Hardened properties of mortar at 400 °C**

| Ratio of walnut (%) | Mix ID | Dry density (kg/m³) at 28 days | Compressive strength (MPa) | Loss in strength (%) |
|--------------------|-------|--------------------------------|---------------------------|---------------------|
| 0                  | R10   | 1960                           | 34.93                     | 17.98               |
| 10                 | B11   | 1756                           | 18.35                     | 39.97               |
| 20                 | B12   | 1528                           | 17.41                     | 15.36               |
| 30                 | B13   | 1392                           | 3.21                      | 46.76               |
| 0                  | R20   | 2028                           | 46.13                     | 18.11               |
| 10                 | C21   | 1820                           | 20.45                     | 30.02               |
| 20                 | C22   | 1640                           | 8.69                      | 35.62               |
| 30                 | C23   | 1396                           | 2.24                      | 68.36               |

**Table 9. Hardened properties of mortar at 600 °C**

| Ratio of walnut (%) | Mix ID | Dry Density (kg/m³) at 28 days | Compressive strength (MPa) | Loss in strength (%) |
|--------------------|-------|--------------------------------|---------------------------|---------------------|
| 0                  | R10   | 1940                           | 22.05                     | 48.22               |
| 10                 | B11   | 1708                           | 11.83                     | 61.3                |
| 20                 | B12   | 1488                           | 4.87                      | 76.32               |
| 30                 | B13   | 1392                           | 2.09                      | 65.33               |
| 0                  | R20   | 1340                           | 38.35                     | 31.92               |
| 10                 | C21   | 1784                           | 13.33                     | 54.37               |
| 20                 | C22   | 1588                           | 5.8                       | 57.05               |
| 30                 | C23   | 1196                           | 0                         | 100                 |
Figure 13. The decreasing of density with temperature for first series.

Figure 14. The decreasing of density with temperature for second series.

3.3.2. Compressive strength

Tables 8 and 9 present the values and reduction of compressive strength after exposure to elevated temperature at 400 and 600 °C then graphically demonstrated in Figures 15 and 16, respectively. It can be seen that the reduction in compressive increased with an upsurge in the ratio of GW, and this reduction is more for the second series when compared with the first series. Different specimens after testing are shown in figure 17. This upsurge in decreasing compressive strength could have resulted from the existence of a lot of voids when the ratio of GW increased and losing GW’s strength after exposure to a high temperature which led to the loss of the bond between the hydrated cement and burned GW particles.
Figure 15. Reduction of compressive strength with exposure to high temperature for first series.

Figure 16. Reduction of compressive strength with exposure to high temperature for second series.
4. Conclusion
Based on the findings of this study, we concluded that GW can be successfully used as a sand replacement in cement mortar production, and the results are summarized below:

- The compressive strength and density decreased by using GW and this reduction was more when the GW ratio increased and the water-cement ratio was less.
- The maximum GW replacement level was 20 and 10% for w/c mixtures of 0.5 and 0.4, respectively, giving compressive strengths greater than 20 MPa and suitable for structural purposes.
- For both series, the mixtures contained more than 20% GW and were considered lightweight mortar.
- The workability decreased sharply when the GW ratio increased and the reduction increased with decreasing w/c.
- The dry density was reduced by increasing the usage of GW in the mixes and the reduction ratio increased when the mortar samples were exposed to high temperatures, and this reduction was slightly more for the second series.
- The use of GW led to more reduction in compressive strength when exposed to high temperatures, and this reduction was more for the second series.
- The use of GW up to 10% for mortar exposed to an elevated temperature of 400 °C can be recommended and is suitable for structural purposes.
- Based on the results, we concluded that it is possible to use GW successfully in the production of mortar with 20% not exposed to elevated temperatures and 10% exposed to temperatures up to 400 °C, which is suitable for structural purposes.
- This investigation can be considered as environmentally friendly and recycling waste materials in mortar production.

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