Ultra-Fine Treated and Untreated Walnut Shell Ash Incorporated Cement Mortar: Properties and Environmental Impact Assessments

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ABSTRACT

The utilization of walnut shell (WS) as partial replacement of aggregates and cement is getting more attention in modern research on construction material. Though WS provides lightweight concrete, but less compressive strength and porous structure of concrete is still problematic for its utilization. Thus, this study is using untreated walnut shell powder (UWSP) and ultra-fine treated walnut shell ash (UFTWSA) as a substitute for cement. Different weight fractions of UWSP and UFTWSA (5, 10, 15 and 20%) were used to investigate the influence on fresh and hardened properties of cement mortar. Further, the durability of all mixes was evaluated by immersing them in different concentrations of MgSO₄ (5, 10 and 20%). The experimental results revealed that the inclusion of UWSP and UFTWSA reduces the fresh and hardened properties of cement mortar. Moreover, UWSP addition has more negative impact on fresh and hardened properties of cement mortar as compared to UFTWSA. The hardened properties of mortar specimens remarkably decreased by immersing in MgSO₄ solution. However, all mixes contained UWSP and UFTWSA and not exposed to acid attack achieved more than 30 MPa and can be classified as good type mortar depending on obtained ultra-sonic plus velocity values (UPV).

1. INTRODUCTION

The urbanization of constructional developments is consuming huge amount of cement and other materials. The cement industries are consuming higher amount of energy during cement production. On the contrary, these industries are contributing massive impacts on the environment by means of CO₂ and other greenhouse gases generation. Therefore, the engineers are focusing for reducing the utilization of cement, in order to protect the environment and saving some cost and energy. In this regard, numerous studies recommended the usage of wastes and considered wastes as an alternative solution for preventing the excessive usage of cement [1-6]. These wastes have been used as partial replacement of cement and or fully / partial replacement of aggregates [7]. The major advantages of using wastes are to saving construction cost, energy, and conservation of natural resources. Additionally, utilization of wastes reduces the negative impacts through proper disposal and enhances the protection of environment. Generally, two common wastes: industrial and agricultural wastes have been utilized for the production of cement mortar. The wide range of industrial wastes including fly ash (FA), silica fume (SF), coal bottom ash, and copper slag [8, 9], recycled plastics [10], recycled waste glass [11, 12] have been used and researched for cement mortar. Whereas, the agricultural wastes composed of list of materials, and considered as promising alternatives for aggregates in cement [9, 13-16].

Numerous researches have been conducted to investigate the influence of agricultural wastes on the concrete properties [7]. The number of agricultural wastes’ investigated is not limited to groundnut shell [17-19], wood ash [20], oyster shell [21], rice husk [22-24] and many others. Among these agricultural wastes, walnut shell is rarely researched yet, and even if any research done on it, is considered walnut as partial fully replacement of aggregates. Nwofor and Sule [17] used groundnut shell ash (GSA) to partial replace ordinary Portland Concrete (OPC). The specimens were prepared by using GSA up to 40% with an increment of 10%, density and compressive strength decreased by increasing GSA content. However, 10% of GSA provided optimum results for these properties, and considered for production of sustainable concrete. Sada [19] used groundnut shell (GS) as 0, 5, 15, 25, 50 and 75% by volume to replace fine aggregates of the concrete. The results showed that density and compressive strength decreased by increasing GSA content. However, 10% of GSA provided optimum results for these properties, and considered for production of sustainable concrete. Husain et al. [25] used 10, 20 and 30% of walnut shell slag (WSS) as partial replacement of fine aggregates for concrete production. The results revealed that the use of WSS caused reduction in strength of concrete. However, the optimum value of compressive strength was obtained as 29.3 N/mm² for 20% of WSS in concrete cured at 28-days. Kamal et al. [7] used WS
as partial replacement of fine aggregates of concrete. The central motivation of their research work was to investigate the potential of utilization of WS for concrete structural application, minimize of waste landfill, and concrete properties. The experimental design was modeled by response surface methodology, and used water to cement ratio (w/c) of (0.38-0.52) and WS (1.72-58.28) by cement weight percent. The result from response surface analysis revealed that WS has significant effect on compressive strength and density of the concrete. Further, the utilization of WS up to 30% for aggregates' replacement with w/c of 0.38 is best suited, and provided acceptable compressive strength for concrete structural application. These results indicated that WS can be potentially used for producing structural and environmental friendly concrete. Hilal et al. [26] used 5 to 50% with each interval of 5% of walnut shell (WS) (by volume) and replaced with coarse aggregates for fabricating self-compacting concrete (SCC). The laboratory results revealed that the use of WS reduced the fresh and hardened properties of SCC. Further, the optimum values of slump flow diameter, compressive and bonding strengths were obtained as 560 mm, 35 MPa and 6.55 MPa respectively at 35% of WS. As based on their results, 35% of WS was considered to obtain lightweight SCC. Hilal et al. [27] used 5 to 25% of crushed WS (CWS) to partial replace the coarse and fine aggregates for the production of environmental concrete. The results revealed that the use of CWS caused to reduce all tested properties (compressive strength, flexural strength, splitting strength and dry density) reduced except water absorption at 28-days curing age. Further, 15% of CWS provided the optimum values of these properties.

Mohammed et al. [28] conducted experimental research on using WS as partial replacement of fine aggregate by ratios (10, 20, and 30) in cement mortar. They tested density, workability, and compressive strength at ages of 7, 14, and 28 days. Also, they found the influence of the applied temperature (400 and 600°C) on the compressive strength. their results showed all investigated properties decreased by utilizing WS and 20% of WS with a water-cement ratio of 0.5 gave a lightweight- structural mortar if exposed or non-exposed to 400°C. Where, WS takes years to decompose, and due to its high hardness, it is used for the rough surfaces of concrete floors, masonry walls, and shotcrete surfaces. Further, WS has unique properties and is considered an environmentally friendly alternative material for use in concrete. Also, based on the discussions from previous research, WS can be used for the production of lightweight concrete.

The previous paragraphs show that there is no use of WS as a partial replacement of cement. Therefore, this study investigates the effectiveness of utilizing untreated walnut shell powder (UWSP) and ultra-fine treated walnut ash (UFTWSA) in different amounts (5, 10, 15, and 20%) by weight to replace ordinary Portland cement. The workability, fresh density, water absorption, dry density, loss in weight, and compressive strength of the cement mortar were calculated at ages of 7, 28, and 90 days. Also, the effects of different MgSO₄ solution concentrations on the durability of the cement mortar contained UWSP and UFTWSA were studied.

2. MATERIALS AND METHODS

2.1 Materials

OPC having specific gravity value of 3.15 kg/m³ and value of 0.380 m³/g for the specific surface area. Normal sand with maximum size of 2.36 mm and a value of 2.68 kg/m³ for specific gravity was utilized as fine aggregates and the grading as shown in Figure 1. The tap water was used for curing at first stage. The UFTWSA with an average range of particle size of from 17.86 to 13.40μm with a specific surface area of 1.686 m²/g and a specific gravity of 2.7 kg/m³ were used for this research.

![Figure 1. Grading curve for Fine aggregate](image)

2.2 Preparation of UFTWSA

At the beginning regular WS waste dried in an electric oven at 105 ± 5°C for 24 hours to remove any moisture. Afterwards, these WSs were grounded through milling for 3 hours to reduce particle size and increase surface area. This action for WSs can improve the pozzolanic reactivity and provide untreated walnut shell powder (UWSP). The UWSP burnt at 700 °C in an electric furnace for 2 hours to obtain treated walnut shell ash (TWSA). Finally, TWSA was grounded again by using electric powered grinder for 3 hours to produce UFTWSA. Figure 2(a) and (b) show UTWSA and UFTWSA respectively.

![Figure 2. Walnut shell powdера](image)

2.3 Details of the mixtures

Ten mixtures of cement mortar were used in this study in order to observe the effects of UWSP (series I) and UFTWSA (series II) inclusion on the mechanical and physical characteristics of the mortar. The different percentages of UWSP and UFTWSA (0%, 5%, 10% 15% and 20%) by weight used as replacement of cement. Table 1 shows the design of mixture, and 189 cubes were prepared for the experimental testing.
2.4 Testing methods

The slump flow diameter of the mortar was calculated according to ASTM C1437 standard [29]. The fresh mortar was filled in mini steel cone and lifted carefully on a flow table. The used flow table was dropped for 25 times in 15 seconds until a steady state of the mortar was reached. The average value of the fresh mortar diameter was calculated at two random locations, as shown in Figure 3.

Figure 3. Slump flow test (a) control mix (b) M-5% UWSP (c) M-5% UFTWSA

Further, the fresh and dry density values were calculated according to BS EN 1015 - 6 [30], the ASTM C597-09 [31] standard was used to calculate ultrasonic pulse velocity (UPV) at 90-days curing age. Moreover, the compressive strength was measured by using 50 mm cube size according to BSI EN 12390-3 standard [32] at 7, 28 and 90 days curing age. The compressive strength value was recorded on the basis of average of three specimens. And in accordance with the ASTM D5229 [33] standard, the water absorption test was carried for mortar. For water absorption test, the mortar specimens at 90-day curing age were dried through oven for 24 hours, and weight was measured. Subsequent to this action, the mortar specimens were immersed in water for 48 hours, and weight was measured. The absorbed moisture was calculated through difference in weight between “oven-dried” and “fully wet” specimens.

The acid attack test was also carried to examine the persistence and resilience to acidic chemicals for the mortars. For this test, the total number of 81 cubes of size 50 mm was casted and divided as: three cubes for reference mortar specimen and three for each percentage of UWSP and UFTWSA mixes. All these 81 specimens were cured in water tank for 30 days, and dried at room temperature for 1-day, and weight was calculated for specimens. Subsequent to it, all these specimens were submerged in different percentages (5, 10 and 20%) of chemical solution of magnesium sulfate acid (MgSO₄) for 60 days as shown in Figure 4.

The percentages of MgSO₄ were as per weight of the water to maintain pH level of 3 in order to formulate the solution.

The care was taken to maintain pH of the chemical solution. The mortar's cubes were soaked for 60 days in the acid then removed to air dry at room temperature for one day before being tested for compressive strength. The mortar's resistance to acid attack has been established based on percentage of loss for weight and compressive strength’s when contrasted to the cubes which were treated in plain water.

Figure 4. Curing mortar specimen in magnesium sulfate acid (MgSO₄) solution

3. RESULTS AND DISCUSSION

3.1 Fresh properties

3.1.1 Sump flow and Fresh density

Table 3 presented the results of slump flow and density of the fresh mortar. The slump flow and fresh density of each control, UWSP and UFTWSA mixes were measured through a simple flow table test prior to casting. Figure 5 depicted the slump flow results of each control, UWSP and UFTWSA mixes. The slump flow for both UWSP and UFTWSA mixes was reduced gradually by increasing their respective content. The slump flow for Series-I reduced from 180 mm to 120 mm for 5% to 20% of UWSP. Whereas, the slump flow reduced from 210 mm to 180 mm for 5% to 20% of UFTWSA (Series-II). These values indicated that UFTWSA provided better workability as compared UWSP. This is the reason that the maximum ratio for reducing the slump value was obtained as 45% for mix UWSP20 and for UFTWSA obtained as 18.2% for mix UFTWSA20. The reason of the reduction in the workability may be caused by the high demand of water by UWSP as compared to UFTWSA, which resulted from their higher absorption ratio of water [26]. However, based on reduction for both UWSP and UFTWSA mixes, it can be concluded that both (UWSP and UFTWSA) have higher water absorption as compared to ordinary cement.

<table>
<thead>
<tr>
<th>Series</th>
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<th>Cement</th>
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<th>UFTWSA</th>
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<th>Water</th>
</tr>
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<tr>
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<td>0</td>
<td>750</td>
<td>250</td>
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<td>25</td>
<td>750</td>
<td>250</td>
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<tr>
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<td>50</td>
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<td>75</td>
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<td>UFTWSA20</td>
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<td>100</td>
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Table 2. Slump and Fresh density of mortar

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<tr>
<th>Series</th>
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<th>Slump (mm)</th>
<th>Reduction in slump %</th>
<th>Fresh density (kg/m$^3$)</th>
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<td>-</td>
<td>2400</td>
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<td></td>
<td>UFTWSA20</td>
<td>180</td>
<td>18.18</td>
<td>2230</td>
</tr>
</tbody>
</table>

Figure 5. Slump flow for control, UWSP and UFTWSA mixes

Figure 6. Fresh Density for Control, UWSP and UFTWSA Mixes

Figure 7. Reduction in workability of control, UWSP and UFTWSA mixes

Table 3. Dry Density and Compressive Strengths of control, UWSP and UFTWSA mixes

<table>
<thead>
<tr>
<th>Series</th>
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<th>Compressive strength (MPa)</th>
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<td></td>
<td>7 day</td>
<td>28 day</td>
</tr>
<tr>
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<td></td>
<td>UWSP5</td>
<td>2300</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td>UWSP10</td>
<td>2200</td>
<td>33</td>
</tr>
<tr>
<td></td>
<td>UWSP15</td>
<td>2100</td>
<td>30</td>
</tr>
<tr>
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<td>39</td>
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<tr>
<td></td>
<td>UFTWSA5</td>
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<tr>
<td></td>
<td>UFTWSA20</td>
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<td></td>
</tr>
</tbody>
</table>
3.2 Hardened properties

The values of dry density and compressive strength of the control, UWSP and UFTWSA were measured as per standard methods as described previously. Table 3 shows the dry density results and compressive strengths for cubic samples with dimension 5*5*5 cm at 7, 28 and 90 days curing age for control, UWSP and UFTWSA mixes.

![Figure 8. Dry densities for control, UWSP and UFTWSA mixes](image)

Figure 8. Dry densities for control, UWSP and UFTWSA mixes

![Figure 9. Reduction in Fresh and Dry densities for UWSP and UFTWSA mixes](image)

Figure 9. Reduction in Fresh and Dry densities for UWSP and UFTWSA mixes

3.2.1 Dry density

Figure 8 shows the dry density of the control, UWSP and UFTWSA mortar specimens. The results revealed that dry density decreased by increasing the content of UWSP and UFTWSA in cement. Moreover, UWSP provided higher reduction in dry density results as compared to UFTWSA addition in cement mortar. The maximum reduction of 15.3% in dry density was obtained for UWSP20 mix. The main reason of reduction in dry density of the mixes is due less hydration of the aggregates including walnut shell powder and ash with the cement. As Adebara et al. [34] concluded that the reduction in density values of the concrete mixed with groundnut shell ash and dust is suggesting less hydration with cement. The other reason of such reduction is due to lower specific gravity value of the UWSP and UFTWSA (2.7 kg/m³) as compared to cement (3.15 kg/m³). Further, the reduction of density of the cement mortar helps to reduce the weight of the concrete [35]. Also the Hilal et al. [26] stated that the shape of WS’s formed voids and porosities in the surface of concrete, and due to that the density of the concrete also decreased. This is evident that UFTWSA has less reduction of density of the cement mortar as compared to UWSP mixes.

![Figure 10. Compressive Strength for Control, UWSP and UFTWSA mixes](image)

Figure 10. Compressive Strength for Control, UWSP and UFTWSA mixes

3.2.2 Compressive strength

Figure 10 shows the values of the compressive strength of control, UWSP and UFTWSA mixes of the mortar. The results revealed that the compressive strength decreased by increasing the content of both mixtures (UWSP and UFTWSA) for all curing age. Further, the inclusion of UFTWSA produced better compressive strength performance as compared to UWSP inclusion. The main reason is of the higher contact surface area of the UFTWSA with the cement as compared to UWSP [26]. On the other, the inclusion of UWSP caused to have voids and porous structure for the mortar due to its shape and size [27], which weakened the resistance to applied forces on the specimens. Further, the gradual reduction in compressive strength for UWSP and UFTWSA addition as compared to control mix is due to weak bonding between cement and mixtures. Additionally, it is also possible that the quantity of cement paste is unable to fill the gaps and voids appeared due to UWSP and UFTWSA addition. Generally, it can be seen that an increase in the proportion of UWSP and UFTWSA caused a reduction in compressive strength, mostly as a result of the reduction in the amount of cement in the cement paste. This may lead to a decline in the quantity of hydration outputs such as belite (C2S) and a-lite (C3S), which are accountable for the increase in the strength and absorbency (reduction in the density) of the blended paste. Alternatively, there may be a decrease in the interlocking between particles of UWSP and UFTWSA, particles of cement and sand, which leads to an increase in the number of voids in the mortar. Also the reduction in compressive strength of the mortar due to addition on UWSP and UFTWSA is suggesting for the production of lightweight concrete [16], and are preferable for low stress structural components like: mortar, mass concrete and floor screed [34]. Moreover, Sada et al. [19] suggested that WS significantly affect the weight and strength of the concrete and cannot be used for production high strength concrete exposed to water.
Further, the compressive strength of control, UWSP and UFTWSA mixes increased with increasing curing age, which indicated that curing age has a significant effect on compressive strength [26, 34, 35]. The maximum value of compressive strength was obtained as 44 MPa and 45 MPa for UWSP5 and UFTWSA5 mixes respectively at 90-days curing age. As based on these results, it can be remarked that UPWS and UFTWSA addition is suitable for producing low strength concrete, however, upper duration curing age can provide higher compressive strength of the concrete.

3.2.3 Influence of MgSO₄ solution compressive strength and density of mortar

The concrete cubes after 90-days curing were immersed in MgSO₄ solution, and were examined for dry density and compressive strength of the mortar. Three different solutions of MgSO₄ (5, 10 and 20%) were prepared to investigate the influence of alkali acidic attack on hardened properties of the mortar. Table 4 shows the results of dry density and compressive strength of the mortar subjected to different solutions of MgSO₄.

![Figure 11. Influence of MgSO₄ Solution on Dry Density of Mortar](image)

Figure 11 shows the results of dry density values of the mortar specimens immersed in MgSO₄ solution. The results revealed that the MgSO₄ solution concentration has a negative effect on the dry density of the mortar specimens. The dry density values were decreased by increasing the concentration of MgSO₄ solution. Similarly, UFTWSA10 recorded small increase in density values at 10% as compared UFTWSA5. Beyond that, the density values were decreased by increasing either UFTWSA content or MgSO₄ concentration.

![Figure 12. Influence of MgSO₄ Solution on Compressive Strength of Mortar](image)

Figure 12 represents the compressive strength of control, UWSP and UFTWSA mortar specimens immersed in MgSO₄ solution concentration. The results revealed that the MgSO₄ solution concentration has a significant negative impact on the compressive strength of the mortar mixes. The compressive strength values were decreased by increasing either the concentration of MgSO₄ or UWSP and UFTWSA content. Arafa et al. [36] and Lee et al. [37] also confirmed the reduction of concrete compressive strength by increasing the content of aggregates and solution concentration of MgSO₄. The highest value of compressive strength was obtained at lower content of UWSP and UFTWSA content and lower concentration value of MgSO₄ solution.

3.2.4 Absorption ratio

Figure 13 shows the water absorption for control, UWSP and UFTWSA specimens immersed in tap water and MgSO₄ solution. The mortar specimens cured at 90-days were tested for water absorption test. The results revealed that the control mix has lowest value immersed in either tap water or MgSO₄ as compared to UWSP and UFTWSA. The inclusion of UWSP and UFTWSA resulted in increase the water absorption ratio of mortar immersed in tap water. Further, UWSP had higher negative effect to upsurge this absorption ratio as compared with UFTWSA, which can be turned to the more porous structure of UWSP mixes than UFTWSA mixes. This is mainly because of the higher absorption nature of UWSP due

![Figure 13. Influence of MgSO₄ Solution on Absorption Ratio of Mortar](image)
to size and shape as compared to UFTWSA. Moreover, as it seen earlier that UWSP inclusion caused to produce voids and decrease the cement paste in mortar due to which it absorbed more water may be due to the higher surface area.

Figure 13. Absorption Water for Control, UWSP and UFTWSA for Tap Water and MgSO$_4$ Solution

The immersion of control, UWSP and UFTWSA specimens in MgSO$_4$ led to somewhat change in water absorption ratio; where this variation was altered as increase, or decrease or unchanged with increasing concentration of MgSO$_4$. The highest water absorption ratio was achieved for UWSP20 mix at 20% of MgSO$_4$ which can be explained to higher amount of voids in UWSP20 mix filled with particles of MgSO$_4$ and led to decrease their ability to water absorption.

3.2.5 Weight of mortar specimens

Figure 14 shows the weight of the specimens for control, UWSP and UFTWSA mixes immersed in tap water and MgSO$_4$ solution. The mortar specimens at 90-day curing age were dried through oven for 24 hours, and weight was measured. The results revealed that the weight of the mortar specimens gradually reduced by increasing the content of the UWSP and UFTWSA. The inclusion of UWSP in mortar specimens caused more reduction in weight as compared to UFTWSA addition. It is mainly because of the voids and less quantity of cement paste in mortar. Further, Hilal et al. (2020) also observed reduction in weight of the concrete material due to addition of WS [26]. The reduction in weight of the mortar specimens suggesting that the use of WS can produce lightweight concrete facing lower stresses. Further, Hilal et al. (2020) suggested that utilizing WS can save the cost of the aggregates and or cementitious material when replaced with it [27].

Further, the reduction in weight of the mortar specimens was also observed by increasing the solution concentration of MgSO$_4$. The control and UWSP specimens have higher weight reduction as compared to UFTWSA when tested under MgSO$_4$ solution. Further, a small increase was observed at UFTWSA10 mixes for 10 and 20% of MgSO$_4$ solution. It may be because of the chemical reaction between silicate and cementitious material.

3.2.6 Ultrasonic pulse velocity

The results of UPV for control mortar specimens, and UWSP and UFTWSA mixes are depicted in Figure 15. The inclusion of UWSP and UFTWSA caused reduction in UPV of the mortar mixes, and UWSP offered more reduction in UPV as compared to UFTWSA addition. This reduction can be attributed to porous nature of UWSP and UFTWSA besides increasing of voids that resulted from less workability in mortar. The UPV results for UWSP mixes are confirmed with the dry density, water absorption and weight of the specimens. Generally, UPV is considered for ultrasonic waves pass through different materials such cementitious, aggregates, WS and voids. Also, the reduction in UPV of the mortar is due to that as it partly transmitted and partly reflected through these materials. This is the reason that UPV for mortar specimens mixed with UFTWSA have higher contact surface area, which reflected the ultrasonic pulses and provided higher values of UPV. However, UPV of all tested specimens indicated that mortar still classified as good type.

Figure 14. Weight of the Specimens for Control, UWSP and UFTWSA in Tap Water and MgSO$_4$ Solution

MgSO$_4$ solution has negative impact on UPV results for mortar specimens mixed with UWSP and UFTWSA. The reduction in UPV increased by increasing the solution percentage of MgSO$_4$. The effect of MgSO$_4$ was more worst on mixes contained UWSP compared with UFTWSA mixes, and the lowest value of UPV was 1.9 km/s for UWSP20 immersed in 20% of MgSO$_4$. This high reduction in UPV, especially for mixes contained UWSP, can be attributed to the porous nature of these mixes which saturated with MgSO$_4$ solution and reduce the UPV.

Figure 15. UPV of the Specimens for Control, UWSP and UFTWSA in tap water and MgSO$_4$ Solution

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4. CONCLUSIONS

The possibility of utilization of UWSP and UFTWSA as a substitute for cement in mortar has been investigated. For this purpose, cement mortar mixtures with UWSP and UFTWSA replacement (5, 10, 15 and 20%) were made and the properties in both fresh and hardened states were evaluated, from the experimental results the following conclusions can be drawn:

1. The fresh density mortars’ made by partially replacing cement with UWSP and UFTWSA is decreases.
2. For a given w/c ratio, an increase in the percentage of substitute of OPC with UWSP and UFTWSA decreases the slump value as well as reduces the workability.
3. Generally, it can be concluded that an increase in the proportion of UWSP and UFTWSA means a reduction in mortar compressive strength, mostly as a result of the reduction in the amount of cement in the cement paste. The UFTWSA has less effect on decreasing the compressive strength than UWSP.
4. The compressive strength of mortars in two series almost met the target strength at 28 days.
5. Mortar made with UWSP and UFTWSA is acceptable for various construction works, even though the compressive strength is decreased when using UWSP and UFTWSA as a replacement for cement.
6. Since the addition of UWSP and UFTWSA as a replacement for cement reduces the compressive strength of mortar, it is recommended to use mortar at smaller replacement rates for structures purposes that require high technical demands.
7. The durability of normal mortar is higher than against acid attacks, hence, a mortar incorporating variable percentage of UFTWSA is more durable than the mortar containing UWSP.

REFERENCES