

Original Article

# Recycling of Calcination Waste for use as Cement Replacement in Green Building

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**Abstract** - Waste recycling is a major key to sustainability, as it saves natural raw materials and energy consumption, reduces solid pollutants and greenhouse gases emissions. In this context, this research is a continuation of efforts aimed at utilizing waste to produce sustainable construction material. This study presents an early investigation to utilize the calcination waste as a cement replacement, calcination waste (CW) is a solid waste collected during the calcination process of dolomite and/or limestone. Six mortar mixtures were prepared by replacement cement with 0%, 5%, 10%, 20%, 30%, and 40% of CW. Specimens of each mixture were tested for fluidity, setting time, density, and shrinkage, compressive and flexural strength. In order to evaluate the durability of mortars, another set of specimens were cured in 5%Na<sub>2</sub>SO<sub>4</sub> solution for different ages, then these specimens examined for appearance, weight loss, compressive strength loss, microstructure. The results proved the feasibility of replacing cement with 5% and 10% of CW, where the mortar properties were improved in terms of dry shrinkage, compressive and flexural strength, in addition the losses of weight and strength due to sulfate attack were minor. Otherwise, replacement of cement by CW with 20% or more was affected negatively on the mortar properties.

**Keywords** - Recycling solid waste, sustainability, calcination waste, green cement mortar, strength, carbon emissions.

## I. INTRODUCTION

The solid waste produced in year 2016 was 2.01 billion tonnes, forecasted to be 2.59 billion tonnes/annum in 2030 and 3.4 billion tonnes/ annum in 2050 as shown in Figure 1 [1]. Based on the "Global Waste Management Outlook", only 13.5% of the global solid waste is recycled, while 85% is disposed of in landfills as indicated in Figure 2 [1].

Construction, industrial, and agricultural activities are the main feeders for the solid waste, where they represent 9.15%, 69.3%, and 18.25% of the solid waste respectively [1]. In construction sector, the massive production of cement is the main environmental challenge. Global production of cement is 4.1 billion tonnes in 2020 and expected to be 4.83 billion tonnes in 2030 [2]. Cement industry is responsible for generation about 200 kg of solid waste/ton of cement [3, 4],

meaning that 0.82 billion tonnes/year of solid waste, this solid waste is known as cement kiln dust. Also, cement manufacturing releases about 7.4% of the global CO<sub>2</sub> emissions [5]. In terms of resource consumption, manufacture of each ton of cement consumes 1.5 ton of natural material [6], and 4-5 GJ of energy [7, 8].

Recycling is the effective manner to reduce solid waste, carbon emissions, and land use, in addition saving natural materials, and energy. In this regard, many studies examined the use of industrial and agricultural waste as a cement replacement; a brief description of a commonly waste utilized as a cement replacement is presented in the next subsection.

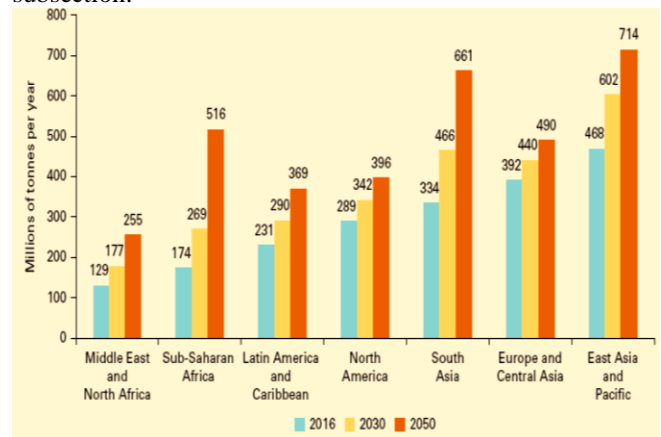


Fig. 1: Global Waste by Region [1]

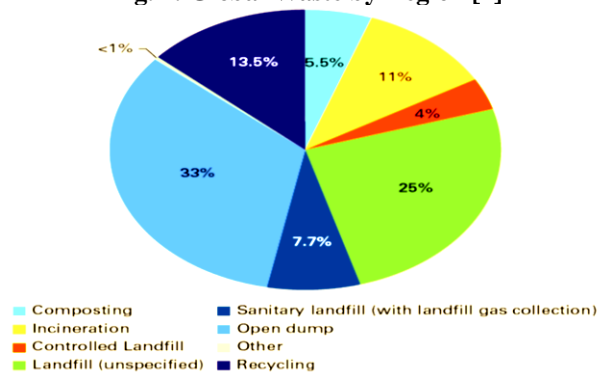
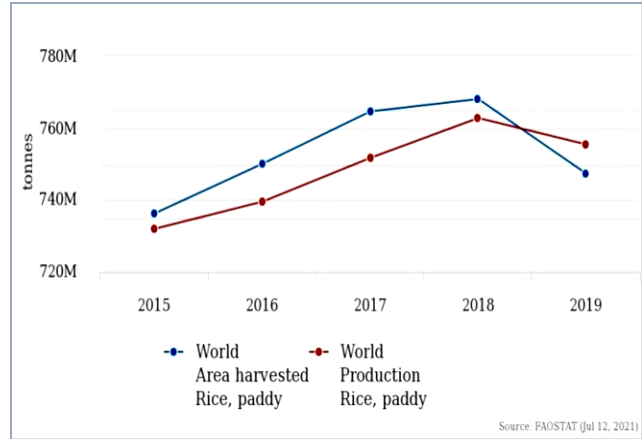


Fig. 2: Global Waste Treatment and Disposal Methods [1]



**A. A commonly waste used as a cement alternative**

Several industrial and agro-wastes have pozzolanic activity which makes it suitable as supplementary cementitious material. Most common waste used as a cement replacement in production of mortar and concrete are; fly ash, silica fume, blast furnace slag, cement kiln dust, clay brick powder, and rice husk ash. Fly ash (FA) is a fine solid waste produced from coal combustion in thermal power plants [9]. About 544 million tonnes produced yearly worldwide [10]. Replacing cement with FA increases density and strength of the cement paste [11, 12, 13]. Silica fume (SF) is a by-product resulting from manufacturing of silicon and ferrosilicon alloys. The estimated global production of SF is 1.5 Mt per year [14]. Extreme fineness of SF and its pozzolanic activity result in dense concrete [15], and improve its compressive, flexural and split tensile strength [16, 17]. Another industrial waste utilized as cement substitution when it grinded in powder form, this waste is the blast furnace slag (BFS). BFS is a solid waste produced from the pig iron manufacturing with annually amount of 300–360 million tonnes globally [18, 19]. Production of one ton of pig iron produces 300 kg of slag, meaning that at least 90 million tonnes per year worldwide. Granulated Blast Furnace Slag is an amorphous material possess cementitious properties, thus replacing cement with BFS produce a (C–S–H) gel, this reduces the pore size and porosity of the cement paste [20, 21] leading to notable improvement in strength and durability of concrete [22, 23]. Many studies have examined using of cement kiln dust as a cement substitute [24, 25] but the high content of alkali, chloride, and sulfate cause deficiency in cement performance [26]. In addition to these materials, clay brick powder which produced from grinding the waste of clay brick into fine particles has pozzolanic activity due to the burning of clay brick at high temperature (600°C- 1000°C) [27, 28]. Several studies reported that replacing of cement with CBP result in a dense and strong hardened cement paste [29, 30]. While; replacement of cement with 25% of CBP reduces the compressive strength of mortar by 25.2% [31]. Moreover, there are many agro-waste consider a feasible source for cementitious materials, where ashes leftover from the burning of some agro-waste often have pozzolanic properties. Several ashes were examined as cement replacement such as; palm fuel oil ash, bagasse ash, bamboo ash, and rice husk ash (RHA) [32]. Among these ashes, RHA is the most widely used in mortar and concrete. RHA is a solid waste produced from rice husk combustion. The worldwide production of paddy rice is 755.5 million tonnes in 2019 as shown in Figure 3 [33], each ton of them gives 200 kg of husk, which converted to 40 Kg of ash on burning [34], meaning that almost 30.22 million tonnes of RHA generated annually. The feasibility of RHA as cement replacement is due to its high content of amorphous SiO<sub>2</sub>, as well as the large surface area of its particles [35].



**Fig. 3: Global Production of Paddy Rice [33]**

**B. Calcination Waste (CW)**

The calcination process includes raising the temperature of substance below its melting point to remove the impurities and improve its chemical reactivity. The calcination process is often done in a horizontal rotary kiln inclined towards the discharge opening as shown in Figure 4.

The most widely used calcined materials in chemical and industrial purposes are dolomite, limestone, and kaolin.

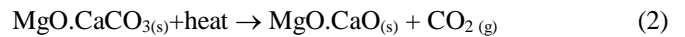
Dolomite, CaCO<sub>3</sub>.MgCO<sub>3</sub> is a sedimentary rock composed theoretically of 54.3% calcite (CaCO<sub>3</sub>) and 45.7% magnesite (MgCO<sub>3</sub>) [36].

Thermal decomposition of dolomite during the calcination process transforms the carbonate minerals into oxides, in presence of CO<sub>2</sub>, this process occurs in two steps as follow [37]:

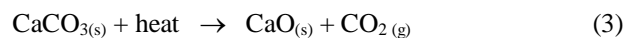
- Magnesite decomposition at 770 °C, according to Equation (1).



- Calcite decomposition at higher temperature of 915 °C, according to Equation (2).



On the other side, Limestone, CaCO<sub>3</sub> is a sedimentary rock composed mainly of calcium carbonate. Calcining of limestone with proper size at a temperature of 1200-1250°C decomposes it to calcium oxide and emits CO<sub>2</sub> according to Equation (3) [38]:



Calcined dolomite and limestone play a major role in many industries such as; refractory materials, steel, paper, glass, fertilizers, sugar refining, cosmetics, paints, and pharmaceutical industries [39].

Calcination waste (CW) is a solid waste in powdered form generated and collected during the calcination process. CW intended here (shown in Figure 5) is a mix of powdered waste collected during the calcination of dolomite and limestone, each of them calcined separately, but the produced waste is collected in the same dump area before being disposed of to the landfill.

The global production of calcined limestone was 20.04 million tonnes in 2019 as shown in Table1 [40], while the produced dolomite was 146.2 million tonnes in 2020, calcined dolomite segment was 39% [41], this means that the global production of calcined dolomite about 57 million tonnes in 2020. Calcination of one ton of limestone or dolomite generates about 2 to 3% of powdered waste according to the quality of quarries, calcination temperature, and efficiency of burning process. This implies that, the global and annum average produced powdered waste from calcination of limestone and dolomite is 5.1 million tonnes, this waste often disposed of in a landfill.



**Fig. 5: Calcination Waste**



**Fig. 4: Horizontal Rotary Calcination Kiln**

Based on a review of the available literature, there is no study related to utilization of CW in a feasible application. Therefore, the novelty of this study is represented in recycling waste generated from calcination of dolomite and limestone and utilizes it as a cement replacement to produce a sustainable construction material. This study presents an original investigation on recycling the CW to reduce dependence on OPC, consequently, reducing the cement dust, and carbon emissions released from cement industry, as well as saving the natural raw materials and energy, in addition utilizing of CW, and reducing the land use in landfills. In this regard, six mixes of cement mortar with substitution ratios 0%, 5%, 10%, 20%, 30%, and 40% of CW were prepared and examined for fluidity, setting time, flexural strength, compressive strength, dry weight, linear shrinkage, sulfate attack, microstructure and EDS analysis. The obtained results of the later five mixtures were compared to those of the reference mix which contains 100% OPC (i.e. 0% CW).

**Table 1. The top 10 producers of Calcined limestone per ton [40]**

Production Volume						
	Year	2015	2016	2017	2018	2019
Global	Production %	17.05M	17.21M	17.65M	19.57M	20.04M
India	17.38%	2.95M	2.44M	2.36M	3.15M	3.48M
Mexico	13.49%	2.34M	2.43M	2.53M	2.55M	2.70M
China	13.31%	2.20M	2.26M	2.29M	2.48M	2.67M
Argentina	9.51%	1.56M	1.68M	1.68M	1.99M	1.90M
Brazil	7.54%	1.18M	1.23M	1.29M	1.50M	1.51M
Turkey	4.74%	750 K	850 K	1.01M	1.10M	950K
Spain	4.42%	886K	955K	925K	1.09M	884K
United States	4.37%	820K	820K	800K	805K	876K
South Africa	2.55%	353K	323k	446k	474K	510K

## II. MATERIALS AND METHODS

### A. Materials

Recycled calcination waste, ordinary Portland cement (OPC), natural sand, and potable water were used in the studied mixtures. Properties and standard specification of these materials are as follows:

1. Recycled Calcination Waste: the used CW is a white smoke powder of 2.6 specific gravity and surface area of 5480 cm<sup>2</sup>/gm; it was collected and supplied by Nourmetec for building materials and thermal products. Nourmetec produces annually about 1.2 million tonnes of calcined limestone and 0.5 million tonnes of dolomite, with 42,000 ton of calcinations waste, which often disposed of in landfill. The microstructure and EDS analysis of the used CW are shown in Figures 6 and 7 respectively, Table 2 presents the chemical constituents.

2. Ordinary Portland cement: OPC- CEMI-42.5N of 3.15 specific gravity and surface area of 3240 cm<sup>2</sup>/gm complies with level of 100% and replaced by different levels of CW waste in the other five mixtures. The used OPC meets ASTM C150/C150-M standard specification [42]. The chemical oxides of OPC are presented in Table 2.

3. Natural sand: the used natural sand has of 2.6 specific gravity, 2.5 fineness modulus, and complies with standard specification ASTM C33 / C33M – 08 [43].

4. Water: the potable water complies with the specification ASTM C1602/C1602M-18 [44] was used in mixing the dry materials and curing the mortar specimens.

5. Activator: alkaline activator solution prepared by mixing sodium silicate solution (35% wt of Na<sub>2</sub>SiO<sub>3</sub>) and sodium hydroxide solution (50%wt of NaOH) with constant ratio of 2, and alkaline activator to binder ratio of 0.35.

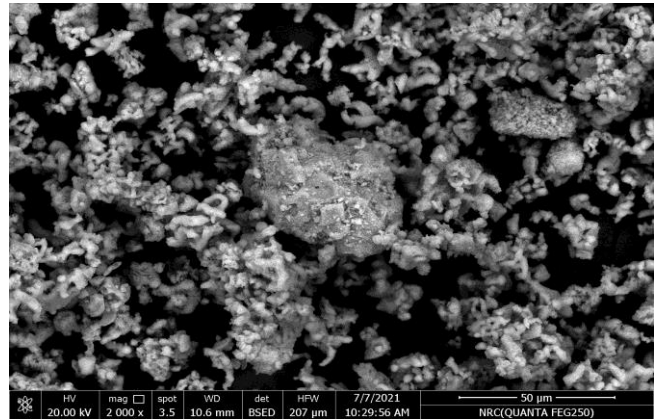


Fig. 6: Microstructure of Calcination Waste

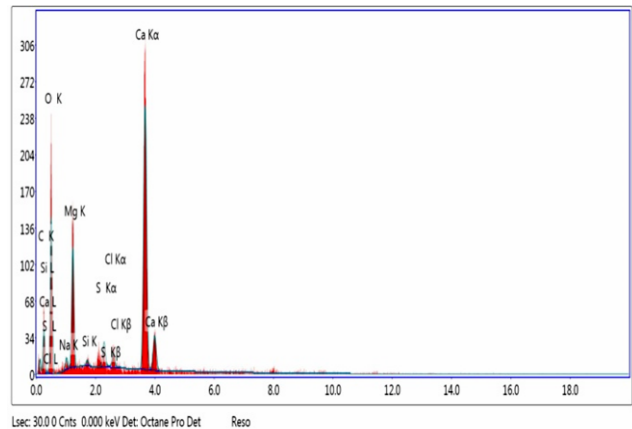


Fig. 7: EDS analysis of Calcination Waste

Table 2. The Chemical Oxides of OPC and CW %wt.

Oxides	SiO <sub>2</sub>	CaO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	SO <sub>3</sub>	MnO
OPC	20.83	62	6.3	3.2	3.3	2.2	0
CW	7.59	50.43	3.48	1.12	17.5	3.4	0.03

### B. Mix Proportions

To evaluate the CW waste as a cement replacement, six mortar mixes were prepared with the proportions presented in Table 3. The water-binder-sand ratio was 0.55-1-3 for all mixes. The reference mixture that contains 100% OPC was named C100, and the mortar mixes composed of different levels of CW 5%, 10%, 20%, 30%, and 40% with OPC were named D5, D10, D20, D30, and D40 respectively.

### C. Samples Preparation and Test Methods

To prepare the mortar specimens, the dry components were mixed with water in electrical mixer to get a homogeneous mixture. Then, the next tests were performed based on the

corresponding standard specifications to evaluate recycling of CW waste as cement replacement.

1. Fluidity: to assess fluidity of fresh mortar, the flow test was executed based on specification ASTM C1437-20 [45].

2. Setting time: setting times of fresh mortars were measured by Vicat apparatus according to ASTM C191-19 [46].

To assess the hardened properties of the investigated mortars, the compressive strength, dry unit weight, flexural strength, and linear shrinkage tests were conducted.

3. Compressive strength test : specimens of 70x70x70 mm were used to measure the compressive strength of mortars according to ASTM C109 / C109M-20b [47].The hardened specimens were cured in water for 7,28,and 56 days and dried before testing.

4. The flexural strength test: it was performed on specimens of 160x40x40 mm after curing for 7, 28, and 56 days according to ASTM C348-21 [48].

**Table 3. Mix proportions (kg/m<sup>3</sup>)**

Mix	OPC	CW	w/b	Water	Binder	Sand
C100	540	0				
D5	513	27				
D10	486	54				
D20	432	108	0.5	270	540	1618
D30	378	162				
D40	324	216				

5. Linear Shrinkage: prismatic samples of 160 x 40 x 40 mm were prepared and cured in potable water for 72 hrs, then the specimens dried and the linear shrinkage measured at 4,11,18,25, and 56 days according to ASTM C157 / C157M – 17 [49].

6. Durability Assessment: in order to evaluate durability of mortars, another group of molded specimens of 70x70x70 mm were exposed to sulfate attack by submerging them in 5% Na<sub>2</sub>SO<sub>4</sub> solution, these specimens evaluated based on the visual inspection at 90 days, density losses, and compressive strength losses at 7,28,56 days. Furthermore, to study the effect of sulfate solutio on the microstructure of mortar, small piece of mortar at age of 6 months was inspected using SEM and EDS techniques.

**III. RESULTS AND DISCUSSION**

**A. Fluidity of Fresh Mortar**

To examine the workability and consistency of the OPC mortar and OPC blended with CW the Fluidity test was performed. The obtained results of both reference mixture and CW mixtures are presented in Table 4. According to the results, it is obvious that as the CW level increases, fluidity of mortar decrease. Mixtures with 5%, 10%, 20%, 30%, and 40% CW have flow less than that of the reference mixture by 4.2, 13.15,21,29, and 39.5 % respectively. This behavior may be related to the non-spherical and irregular CW particles compared with the spherical OPC particles, thus the inter friction between CW greater than that of OPC particles. Consequently, the water required to achieve the same flow of

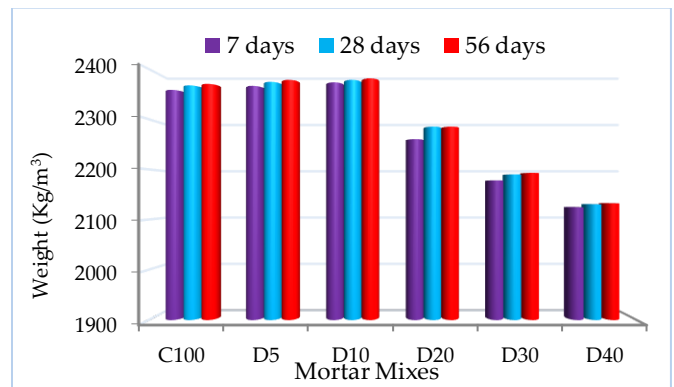
C100 increased with the increase of CW level, otherwise at the same w/c, the mixtures containing CW become dry and resulted in less flow. In general, flow of mortars C100 (100% OPC), D5, and D10 were 111,107,106 mm respectively, which meet with the standard specification ASTM C270-14a [50].

**B. Setting Time**

The recorded setting times of the investigated mortars presented in Table 4. The results indicate that the setting time of C100 (100% OPC) were higher than the setting times of the other mixtures which containing different levels of CW. This means that the higher CW content, the lower setting time. Moreover, the results show that the initial setting time of control mix is higher than that of mixes contain 5,10,20,30, and 40% CW with 4.4, 11.1, 22.7, 29.4, and 33.3% respectively. In addition, the final setting time of C100 is higher than those of D5, D10, D20, D30, and D40 with 10,12,20,26, and 30% respectively. The lower setting time of mixtures contain CW is due to the quick hydration of magnesium oxide and calcium oxide contained in CW [51, 52].

**Table 4. Flow and Setting Time of Mortar Mixes**

Mix	Flow (mm)	I. S. Time (min.)	F. S. Time (min.)
C100	111	180	250
D5	107	172	225
D10	106	160	220
D20	88	139	200
D30	78.8	127	185
D40	66.5	120	175



**Fig. 8: Dry Weight of Mortar Mixtures**

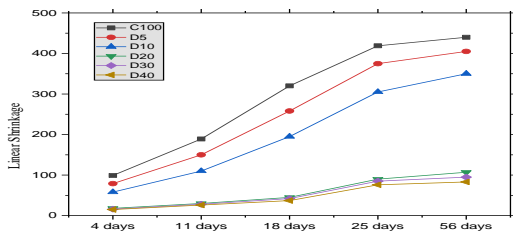
**C. Density**

Density of saturated-surface dry mortar specimens was weighted at 7 d, 28 d, and 56 d. In general, change of density due to replacing OPC with 5%and 10% CW was slight and

lower than 1%. Clearly, the results of this test show in Figure 8 indicate that mortars contain 5% and 10% of CW have density higher than that of the reference mixture with 0.34,0.3,0.34% at 7,28,and 56 days respectively in case of mixture D5,while the increase in case of mixture D10 was 0.3,0.17,and 0.13% at 7,28,and 56 days respectively. This increase likely related to the micro-filler effect of the fine CW particles which leads to refinement of mortar pores system. Otherwise, replacement of OPC with CW levels 20, 30, and 40% reduce the mortar density than the corresponding densities of reference mixture with at 7, 28, and 56 days with 4.15,7.63,and 9.92% at 7 days,3.46,7.46,and10.01% at 28 days, and 3.58,7.46,10.04% at 56 days respectively. This decrease in density of mixtures D20, D30, and D40 is due to excessive of CW more than the interfacial voids in OPC, then the surplus portion of CW displace the OPC. Since, the specific gravity of CW less than that of OPC, the result is a decrease in density.

**D. Linear Shrinkage**

Mortar specimens were tested for the linear shrinkage according to ASTM C157 / C157M – 17 [46] at 4, 11, 18, 25, and 56 days, the test results presented in Figure 9. Generally, the shrinkage of all mortars proportional directly to the specimen age. Also, the observed results indicate that the reference mixture contains 100% OPC (C100) have the higher shrinkage compared to shrinkage of mixtures contain lower levels of OPC. In other words, as the OPC level decreases and CW level increase, the linear shrinkage decrease. Mixture contains 95% OPC and 5% CW(i.e.D5) have shrinkage lower than that of the reference mixture with 20%,21%,19%,11%,and 8% at 4,11,18,25,and 56 days respectively. Also, the mixture D10 (90% OPC with 10% calcinations dust) have shrinkage lower than that of C100 with 41%, 42%, 39%, 27%, and 20% at 4, 11, 18, 25, and 56 days respectively. It was noticed that shrinkage of mixtures contain 20%,30%,and 40% of CW decrease dramatically compared to shrinkage of the reference mixture C100 where mixture D40 have shrinkage lower than that of the reference mixture with 85%,86%,88%,82%,and 81% at 4,11,18,25,and 56 days respectively. The same finding was reported by [53, 54] for mortar contain 7.5% MgO, this finding is due to the fact that the large volume of Mg(OH)<sub>2</sub> resulting from hydration of CW compensates the shrinkage occurred due to OPC hydration.



**Fig. 9: Linear Shrinkage (µm) of Mortar Mixes**

**E. Compressive and Flexural Strength**

The results of 7, 28, 56 days compressive strength are presented in Figure 10. Obviously, the compressive strength of OPC and CW mortars grows with the specimens curing age. The results indicate that replacing OPC by 5% CW results in compressive strength increase at all ages compared to the reference mixture. Compressive strengths of D5 were 34.1, 38.3, and 40.8 MPa at 7, 28, and 56 days respectively; this means that replacement of OPC with 5% CW leads to compressive strength higher than that of reference mixture (30.7,35.9,and 38.5 MPa) with 12.7%,10.58%,and 9.75% at 7,28,and56days respectively. At 7, 28, 56 days, mixture contains 10% CW gave compressive strength of 34.1, 38.3, and 40.8 MPa,which exceeds the strength of C100 with 11%,6.7%,and 5.97% respectively.

The noticeable increase in compressive strength of mortar with replacing cement by 5% and 10% of CW is due to the formation of Mg-S-H in addition C-S-H gel [51], as well as the filler effect of the micro particles of CW [55]. In addition to presence of amorphous SiO<sub>2</sub> in composition of CW with 7.59% as stated in Table 2.

However, the mixtures containing 20%, 30%, and 40% of CW have compressive strength less than that of reference mixture at all ages. Clearly, replacement of cement with 20% CW leads to compressive strength reduction with 2.93%, 4.45%, and 4.55% less than the reduction of C100 at 7,28,56 days respectively. Mixtures containing 30%,and 40% CW follow the same pattern of D20 but with higher reduction values than that of D20.The compressive strength of D30 decreases by 16.9%, 25.3%, and 29.6 % compared to compressive strength of C100 at ages 7, 28, and 56 days respectively, while mixture D40 has compressive strength of 22.3,23.8,and 23.9 MPa at 7,28,56 days respectively, which less than that of C100 with 27.3%,33.4%,and 37.9% at the same curing ages respectively.

Incorporation of 20% or more of CW in mortar causes a compressive strength decrease with a greater rate. This reduction is influenced by the dilution effect due to replacement of OPC with high levels of CW (≥ 20%), consequently, less quantity of SiO<sub>2</sub> which negatively affects on formation of C-S-H and Mg-S-H gel. Hence, the resulting matrix has less strength at all curing times [56]. While, at lower replacement levels (5% and 10%), the dilution effect is compensated by the nucleation and the chemical effects of CaO and MgO, thus the dilution effect disappear and the strength increases [57]. Finally, it can be state that the 28 days compressive strength of mixtures C100, D5, and D10 meets the Egyptian Standard Specification No 2421-1993 [58].

Figure 11 shows the results of the flexural test for the studied mixtures. In general, results of flexural strength test follow the same pattern of compressive strength results. This means that the flexural strength is affected by the same parameters that affect the compressive strength as; dilution effect related

to OPC content, filler, nucleation, and chemical effects of CaO and MgO contained in CW. As shown in Figure 11, the flexural strength of all studied mixtures increases as the curing time increase. The mixture of 100% OPC (reference mixture) attains flexural strength of 7.1, 7.7, and 7.93 MPa at 7, 28, 56 days respectively. Replacement of cement with 5% CW increases the flexural strength by 12.67, 10.65, and 10.1 % more than that of C100 at 7, 28, and 56 days respectively. In similar trend, incorporating 10% of CW in mortar leads to higher flexural strength than that of mortar contains 100% OPC (reference mixture) with 10.98, 6.62, and 6.3% at

7, 28, 56 days respectively. On the other side, the flexural strength decreases with increasing the level of CW to 20% or more compared to the reference mixture. The decrease in flexural strength of D20 is less than the flexural strength of C100 with 3.4, 4.5, and 4.6% at 7d, 28d, and 56d respectively. Obviously, the reduction of flexural strength continues to reach 17.32, 25.45, and 29.6% for D30 at 7, 28, and 56 days. Also, mixture D40 has flexural strength of 5.15, 5.12, and 4.92 MPa with reduction rate of 27.45, 33.5, 37.95 % at 7, 28, 56 days respectively.

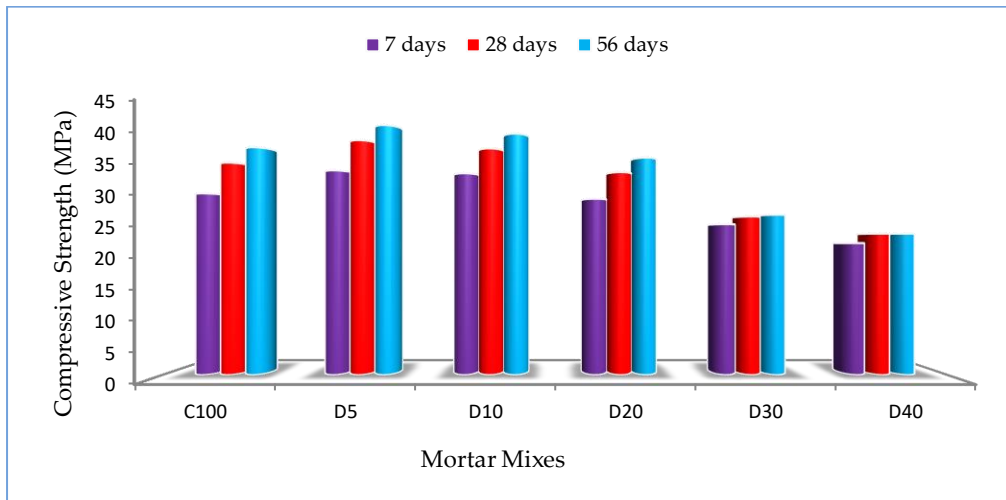


Fig. 10: Compressive Strength of Mortar at 7, 28, and 56 days

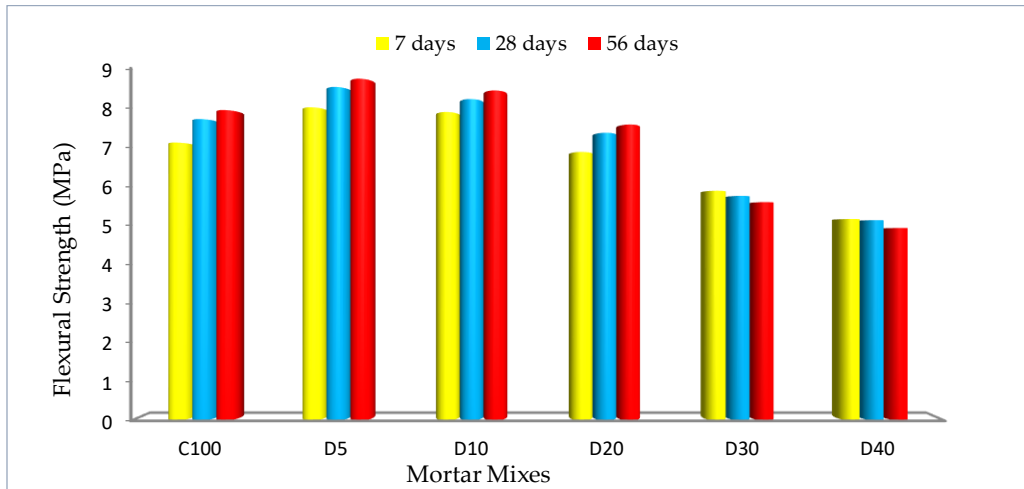


Fig. 11: Flexural Strength of Mixtures at 7, 28, 56 days

**F. Durability (Sulfate RESISTANCE)**

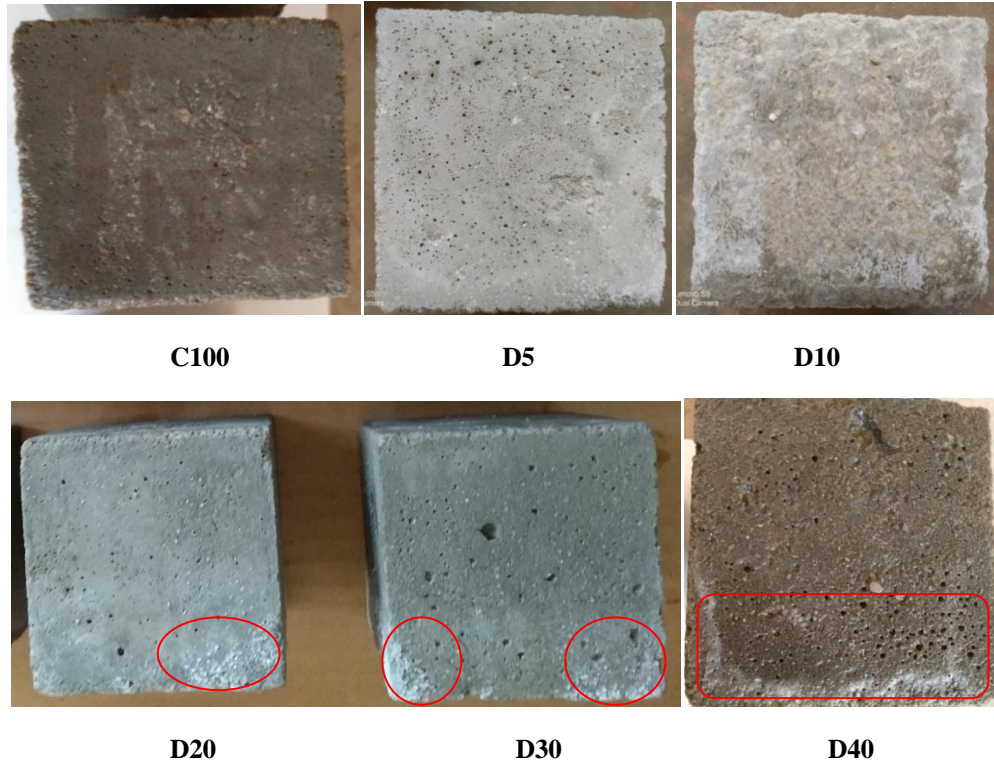
**a) Visual Appearance**

Figure 12 shows the appearance of the mortar specimens after immersion for 90 days in a 5% sodium sulfate solution. After 90 days, it can be seen that no cracks appear on the

surface of any specimen, whether contains 100% OPC or contain calcinations dust. The observed effect of sulfate attack was a whitish precipitate on the surface of CW specimens. The whitish precipitate caused by leaching and deposition of (CaSO<sub>4</sub>) and MgSO<sub>4</sub> formed when the sulfate ions react with Ca (OH)<sub>2</sub> and Mg(OH)<sub>2</sub>. Concerning the

OPC mixture, less white precipitates appears on the surface of the specimen after curing in sodium sulfate for 90 days. This is because the pozzolanic nature of OPC, which presents a dense microstructure of C-S-H gel which leads to high resistance to sulfate [59]. Nevertheless, the amount of C3A

in the hardened OPC specimen reacts with the sulfate ions and forms calcium sulfoaluminate. Subsequently calcium sulfoaluminate react with gypsum ( $\text{CaSO}_4$ ) to produce ettringite.



**Fig. 12: Appearance of specimens cured in 5%  $\text{Na}_2\text{SO}_4$  solution for 90 days**

**b) Weight loss**

Figure 13 shows the recorded weight loss caused by curing the mortar specimens in sodium sulfate for 7, 28, and 56 days. For all mixtures whether containing 100% OPC or containing OPC with CW, the results follow the same pattern after cured in  $\text{Na}_2\text{SO}_4$  solution. The results show that after 7 days of curing in  $\text{Na}_2\text{SO}_4$  solution, the mass of C100 mortar was reduced by 0.16 %, while the CW mortars D5, D10, D20, D30, and D40 loss 0.17%, 0.2, 0.3, 0.47, and 0.68% of their mass respectively. For the same mixture, it can be noticed that the mass loss increase as the immersion time in  $\text{Na}_2\text{SO}_4$  increased. For example, mixture C100 lose 0.16%, 0.17%, and 0.2% of its mass due to immersion in  $\text{Na}_2\text{SO}_4$  for 7, 28, 56 days respectively. Also, as the CW level increase (OPC level decrease) the mass loss increase at all curing times.

**c) The Effect of Sulfate on Compressive Strength**

The compressive strength losses for the mortar specimens caused by sodium sulfate attack compared to compressive strength of specimens cured in potable water presented in Figure 14. The results showed that compressive strength loss for the reference mixture (100% OPC) less than that of mixtures containing CW. Also, the compressive strength losses of both C100 and CW mixtures were slight at early curing age of 7 days, while the losses of all examined mixtures increased at ages of 28, and 56 days. The observed increase of compressive strength at 7 days may be due to the continuity of cement hydration process and the formation of ettringite and gypsum which filling up the fine pores leads to a dense structure, while decreased of compressive strength at later ages is due to increase the expansion effect of  $\text{CaSO}_4$  and  $\text{Mg}(\text{SO})_4$  which results in the formation of micro-cracks and strength loss [60].



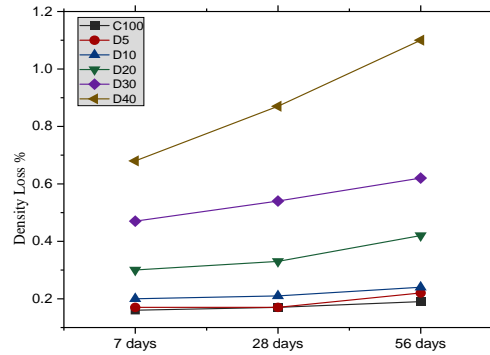


Fig. 13: Weight Loss % at 7, 28, and 56 days

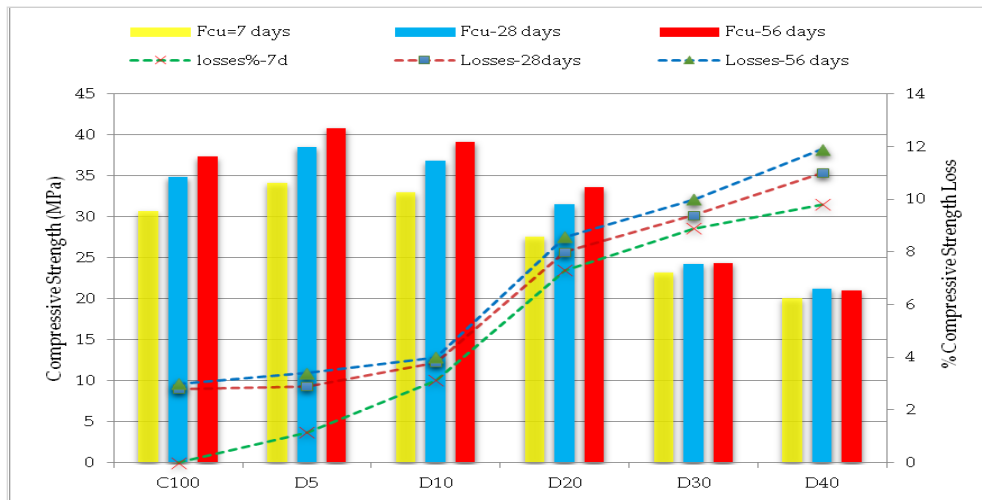
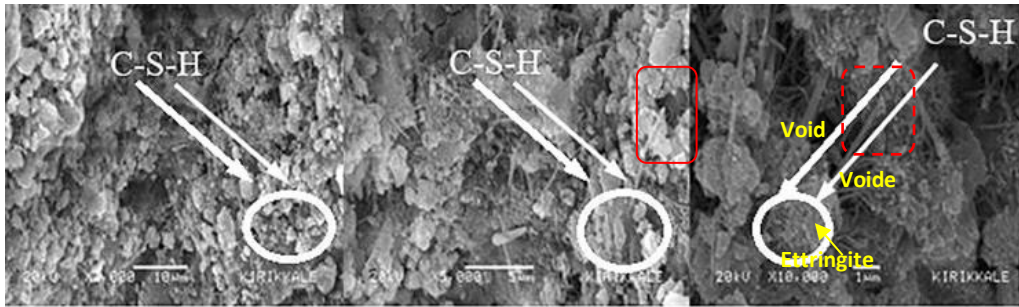


Fig. 14: Compressive Strength Losses % at 7, 28, and 56 days

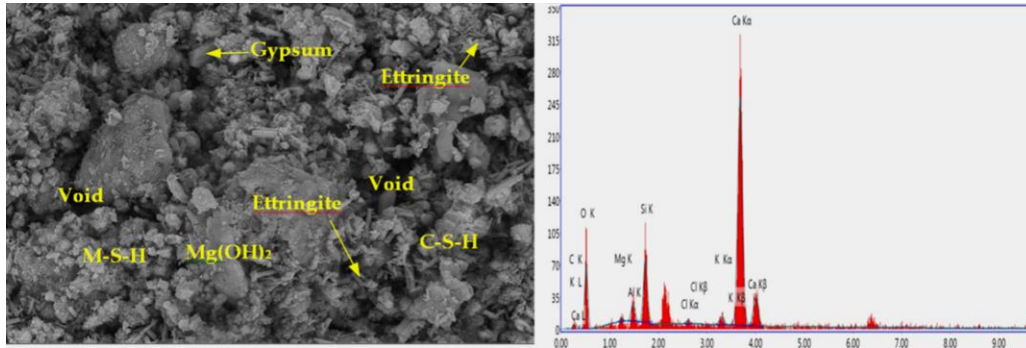
**d) Microstructure and EDS analysis**

As the properties of any cement-based system affected by its microstructure [61], it was helpful to study the microstructure of mortar specimens cured in 5% Na<sub>2</sub>SO<sub>4</sub> solution in order to evaluate their sulfate resistance. SEM images and the corresponding EDS spectrums of mortar specimens after six months of curing in 5% Na<sub>2</sub>SO<sub>4</sub> solution are shown in Figure 15. In general SEM image of reference mortar specimen shown in Figure 15.a [62] indicates that OPC mortar has some needle-like products (ettringite) and large voids between C-S-H matrix. Also, SEM images of CW mortars show that replacement cement with 5 and 10% CW lead to dense structure compared to the reference mortar and mortars with 20, 30, and 40% CW due to the filler effect beside formation of CSH and MSH gels. The dense structure means low porosity and more resistance to sulfate attack in terms of density and strength loss, as well as microstructure degradation [63]. Obviously, mortars of 100% OPC, 5%, and 10% CW, containing more C-S-H gel and less needle-

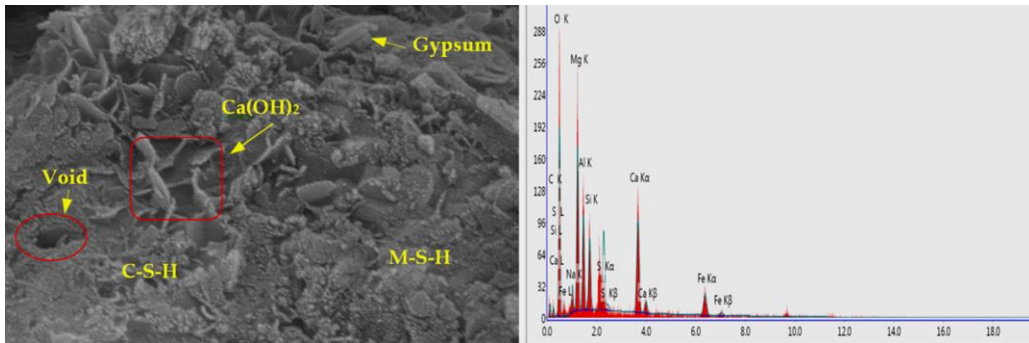
like products (ettringite) compared to that formed in mixtures of 20, 30, and 40% CW. The corresponding EDS results of mortar specimens show that CaO, Si, and O peak and mainly the intensities of Ca, and Si are variable according to OPC and CW levels, where the EDS analysis of CW mortars indicates that as the OPC level decrease (i.e. CW level increase) the higher intensity of CaO and lower SiO<sub>2</sub>. Therefore, replacement of cement with 20% CW or more cause weakens the microstructure as a result of dilution effect and low SiO<sub>2</sub> content. Figures 15.d-e-f show the SEM images of specimens contain 20, 30, and 40% CW, high content of gypsum and larger voids were found beside less amount of C-S-H. The EDS of mortars D20, D30, and D40 show that Si intensity decreased as the level of CW increased. The lower content of Si leads to lower C-S-H and M-S-H gels, which result in weak microstructure and less strength. In addition the content of Ca (OH)<sub>2</sub> increases as CW level increase, consequently, formation of gypsum increase [64].



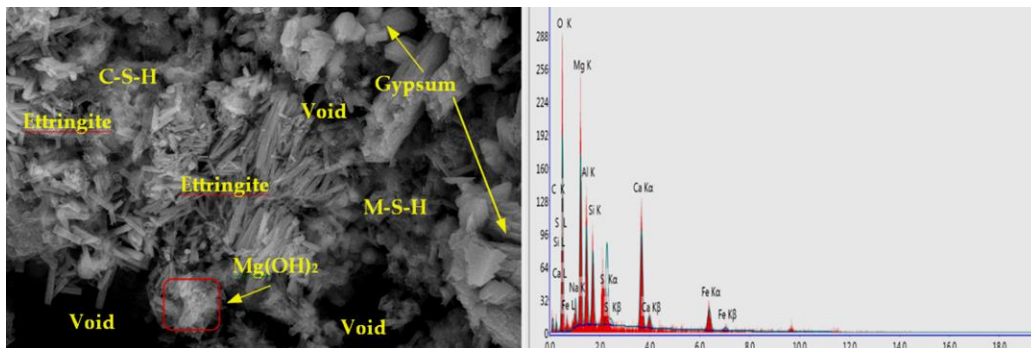
a. Reference mixture [62]



b. D5 mixture

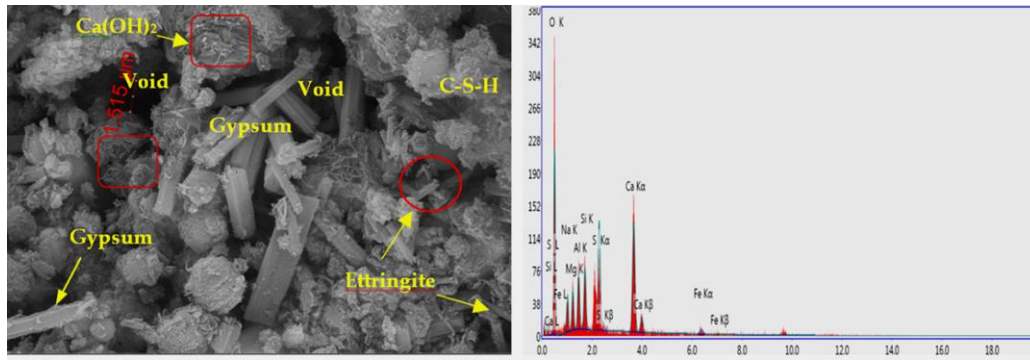


c. D10 mixture

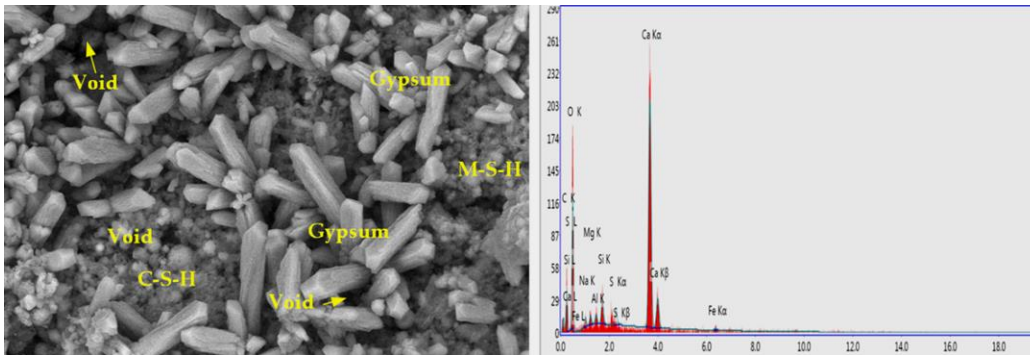


d. D20 mixture





e. D30 mixture



f. D40 mixture

Fig. 15: SEM Images and EDS of specimens cured in 5% Na<sub>2</sub>SO<sub>4</sub> solution for 180d.

IV. CONCLUSIONS

This experimental research was executed to study the utilization of recycled CW as cement replacement in sustainable mortar and concrete. The properties of mortar contain 5, 10, 20, 30, and 40% of CW were studied. Based on the results, the next conclusions can be summarized:

- 1.Replacement of OPC with CW decreases the flow of mortar and increases the required water to achieve a standard consistency. Moreover, the flow of mortar containing 5%, and 10% of CW was complies with standard specification ASTM C270-14a.
- 2.Using CW as cement replacement leads to lower setting time compared to setting time of C100, this is true for all CW mixtures, in any case, the setting time for all mixtures is within the limits of ASTM C191-19 standard specification.
- 3.Mortars including CW have shrinkage strain lower than that of C100 (100% OPC), for example D40 has shrinkage strain lower than that of C100 by 85%,86%,88%,82%,and 81% at 4,11,18,25,and 56 days respectively.
- 4.Replacement of OPC with 5% and 10% CW improves the compressive and flexural strength of mortar compared to the reference mixture; moreover, the attained compressive strength meets the Egyptian Standard Specification No 2421-1993.
- 5.Use of CW with level  $\geq 20\%$  as cement replacement reduces the strength of mortar at all ages compared to C100.

6.Mortar specimens of 5% and 10% CW appear good durability in terms of weight loss and strength loss when immersed in 5% Na<sub>2</sub>SO<sub>4</sub> solution unlike mixes that contain  $\geq 20\%$  CW.

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