



## The Effect of Hydrophobic Amorphous Carbon Powder on the Compressive Strength, Water Absorption and Rheological Attributes of Cement Mortar

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**ABSTRACT:** In this paper, the feasibility of employing amorphous carbon powder as a viable degradation inhibitor for cement mortars made with siliceous aggregates was investigated. Amorphous carbon powder is a by-product of the paraffin industry and was replaced by 0, 4, 6 and 8% of aggregate. Mechanical strength, rheology and water absorption were analyzed considering three common physical factors of concrete namely strength, workability and durability. Mechanical properties of mixtures were obtained using flexural and compressive strengths and rheological attributes were collected through flow table test. Results revealed that adding amorphous carbon powder to the bulk cement mortar could enhance the strength and durability of cement mortar. Replacing 8 wt.% of siliceous aggregate dust filler by amorphous carbon powder caused an increment about twofold in the 28-day compressive strength and reduction of the flow table results by about 20% compared to those of the control mixture. Moreover, hydrophobicity and impermeability properties of amorphous carbon -modified cement mortars, resulted in reduced moisture susceptibility.

**Keywords:** Amorphous Carbon, Cement Composites, Durability, Permeability.

### 1. Introduction

The durability of cement-based composites such as concrete and cement mortar has emerged as an area of focus in recent years (Li et al., 2019). Statistics indicate that rehabilitation of Reinforced Concrete (RC) structures has placed a significant financial burden on authorities' resources. As reported by the Federal Highway Administration (FHWA), \$300 million is

required for either rehabilitation or replacement of US national dilapidated bridges until 2020 (FHWA, 2019). In many cases of which, the RC deck's remediation takes precedence over other treatments (Samadi et al., 2021). Prior to year 2000, it was also estimated that remedial treatments undertaken as a result of chloride-induced corrosion would cost \$5 billion, according to FHWA (Glass and Buenfeld, 2000). Destructive marine environments are

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proved to be efficacious on the durability of concrete due to their vast sulfate and chloride content (Homan et al., 2016; Isteita and Xi, 2017).

Chloride penetration is one of the major potential hazards for the RC structures. Cement matrix provides an appropriate environment for the embedded reinforcements (Ormellese et al., 2006; Glass and Buenfeld, 2000). This is due to an alkaline pH over 12.5 in which the corrosion process ceases to accelerate and causes no more dilapidation (Ormellese et al., 2006; Berke and Hicks, 2004). However, an internal source of unwelcome contamination in mixing progress would derange the passive film around the reinforcements (Berke and Hicks, 2004; Glass and Buenfeld, 2000). On the other hand, the chloride ions rendered by de-icing and marine salt mainly consisting of calcium chloride may contaminate the concrete as an external source (Glass and Buenfeld, 2000; Bamforth et al., 1997). Whether the source of contamination is internal or external, the worst-case scenario is exceeding the Chloride Threshold Level (CTL). CTL is the content of chloride at steel depth in which, the passive film around the steel breaks down and eventually, the corrosion process initiates and gradually worsens (Ann and Song, 2007). Many structures located near water along with off-shore structures (such as parking garage floors, bridge decks and floating docks) have to struggle with these problems (Isteita and Xi, 2017; Homan et al., 2016). New standards have constructors to care about the internal source of chloride ions (Hooton, 2019; Glass and Buenfeld, 2000). Moreover, new technologies such as additive manufacturing give more control on concrete production and have substantially reduced the risk of internal contamination (Li, 2018; Kooshafar and Madani, 2017). Considering the advances in the concrete production, chloride diffusion from external sources must be contained. As a result, several solutions are put forward to cope with this problem. Allen et

al. (1993) suggested resin repair mortars such as epoxy resins as an inhibitor especially in members with a concrete cover less than 12 mm. These mortars have widely been used as coatings. However, statistics show that periodic maintenance of 4 to 15 years is necessary as the coating debonds over time. Tatematsu and Sasaki (2003) used a salt adsorbent to separate and deactivate chloride ions in concrete. Kumar et al. (2018) studied the effect of ground granulated blast furnace slag together with calcium nitrate as an inhibitor.

Another crucial hazard for cement mortars is sulfate attack, which is defined as the physical and chemical reactions between hardened cement paste and sulfate ions. Sulfate attack also may be of an internal or external source. Two main products of the sulfate attack are calcium sulfate and ettringite (Collepari, 2003). Ettringite production is followed by enlargement which exerts pressure on the mortar and causes dilapidation (Ghasemalizadeh and Toufigh, 2020). The latter is also responsible for more permeability. Various methods have been used to prevent or treat the sulfate attack out of which using Ordinary Portland Cement (OPC) types II and V and pozzolanic cement is well-practiced particularly in sulfate dominated environments (Steiger, 2005). Tricalcium aluminate ( $C_3A$ ) as an early-age strengthening agent reacts with the sulfate ions. Type II and IV of OPC have limited components of  $C_3A$  to 8 and 5%, respectively, thus reducing the reaction rate and helping to prevent the sulfate attack. Torii and Kawamura (1994) suggested that the replacement of OPC by fly ash and silica fume effectively enhances the resistance of the mortar to the sulfuric acid and sulfate solution attack. Lee (2009) stated that the use of recycled fine aggregates up to a maximum 50% replacement is effective under severe magnesium sulfate environment, irrespective of the type of recycled fine aggregates.

Water is one of the prerequisites for chloride and sulfate attack (Mobasher,

2006). Homan et al. (2016) illustrated that the moisture content of an RC plays a relatively crucial role in increasing the migration rate of chloride ions, owing to the additional seepage pressure provided by water. Subsequently, the reduction in the amount of absorbed water seems viable to increase the durability against chloride and sulfate attack. For example, the reduction in concrete absorption by addition of nanolime is proven to enhance durability of concrete (Coppola et al., 2018; Taglieri et al., 2017).

De Weerd et al. (2015) stated that although addition of waste glass powder can increase the risk of uncontrolled expansion due to alkali silica reaction, it works as an inhibitor when used as fine aggregate ( $\leq 1$  mm) and in high percentages around 20% to 30%. Another alternative is hydrophobic treatment as an effective technique to minimize moisture transport into concrete. Hydrophobic treatment positive effect on durability and service life has been documented (Mora et al., 2019; Qu and Yu, 2018). As a result, many researchers have striven to prevent water absorption by various means. Zhou et al. (2020a,b) devised a new polymer with a hydrophilic head that absorbs cement hydration products and a hydrophobic chain that can act like a gate, stopping the transportation of ion-carrying liquids such as water. Introduction of 0.9 wt% of inhibitor was shown to have reduced water absorption to 30% of the reference value.

Asipita et al. (2014) deployed green *Bambusa arundinacea* leaves as a natural low-risk inhibitor and observed approximately 70% reduction in the water absorption after 2 hours. They used only 2 wt% of inhibitor and compared its performance with calcium nitrite and ethanolamine. The *Bambusa arundinacea* outperformed both inhibitors. Research conducted by Wong et al. (2015) rendered the effectiveness of paper sludge ash when used in the form of powder as cement replacement and coating. Tittarelli and Moriconi (2011) applied an aqueous emulsion of an alkyl-triethoxy-silan as a

hydrophobic agent. The experiments proved that bulk treatment is more effective during the concrete lifespan and using hydrophobic material as surface treatment is only efficacious during the initial dry-wet cycles.

Mora et al. (2019) developed hydrophobic silica particles which were effective even by low-percent incorporation. The effect of sodium acetate, fluoropolymer, silicone resin and silane when used as a surface hydrophobic inhibitor was assessed and results demonstrated that there is a positive correlation between surface cohesion and hydrophobicity (Liu and Hansen, 2016). Al-Kheetan et al. (2019) investigated whether concrete moisture content affects the performance of sodium acetate, fluoropolymer, silicone resin and silane as surface inhibitors. The results showed that silane is best for dry concrete mixtures while sodium acetate works best when the concrete is saturated.

Amorphous Carbon (AC) powder that is a processed by-product of the paraffin industry, is certified to have hydrophobic attributes (Ziari et al., 2017a,b). Where the AC powder to be disposed of as landfills, it would contaminate the soil and water. On the other hand, the utilization of waste as a construction material is a suitable means of disposal (Habibnejad Korayem et al., 2018; Ziari et al., 2017a,b). Besides, replacing aggregate by AC can help to reduce natural resource depletion (Madani et al., 2016). If applied, AC will be one of few recycled inhibitors that have low financial and environmental cost and can be easily added to bulk concrete. This is an advantage compared to surface inhibitors. Habibnejad Korayem et al. (2018) have investigated the effectiveness of AC in rutting and fatigue performance of asphalt mixtures. This research demonstrated that AC has sufficiently enhanced the moisture resistance properties of the asphalt mixture. Consequently, and due to the similarities between asphalt cement and cement mortar, AC powder to be investigated for

enhancement in concrete's mechanical and durability attributes. In the current study, AC is used as a partial replacement of aggregates to achieve a more durable mortar. The mechanical and rheological characteristics of concrete with and without AC were compared. To measure the influence of AC powder on durability, specimens were exposed to water absorption test. To detect the mechanical and rheological features, flexural strength, compressive strength and flow table were performed. The results indicate a significant increase in compressive strength and better performance of AC-modified concrete in moist ambiance. Conclusively, AC powder is an appropriate inhibitor due to its cost, environmentally friendly features and easy use. The data from this research can be used by stakeholders and policymakers to impose appropriate measures in selection and preparation of concrete materials in future constructions.

## 2. Materials

The specific gravity and water absorption for the utilized silica aggregate were 2.73 and 0.5%, respectively. The aggregate was graded in compliance with BS 196-1 (2005) and portrayed in Figure 1.

OPC Type II from Tehran Cement Plant was used as the binder. The X-Ray Fluorescence (XRF) results were used to identify the chemical compositions of OPC

which is depicted in Table 1. The AC powder which was used as a partial replacement of aggregates is a solid waste of the paraffin industry. Also, Table 2 indicates the physical and chemical properties of AC powder.

Figure 2 shows the visual and Scanning Electron Microscopy (SEM) images of the AC powder passed sieve #200. The surface roughness of AC is vividly seen in Figures 2b-2d at micro and nanoscale.

Experimentally, it was proved that using Polycarboxylate Ether (PCE) superplasticizer is best for AC dispersion (Shahbazi et al., 2020). As a result, PCE was utilized at 0.1% of cement mass for all mix designs.

## 3. Methods

Mixing was done as outlined in BS 196-1 (2005) with some exceptional changes. Hobart mixer equipped with a 5-liter bowl was used for mixing mortar. Mixing started with 30 seconds of dry mixing of sand, cement and AC powder to help the uniform dispersion of AC with other solid components. This stage was followed by adding the water and PCE superplasticizer in the next 30 seconds at a relatively constant rate. Then, 1 minute of medium speed mixing was followed by 75 seconds of rest. The final stage was mixing for a minute at a medium speed.

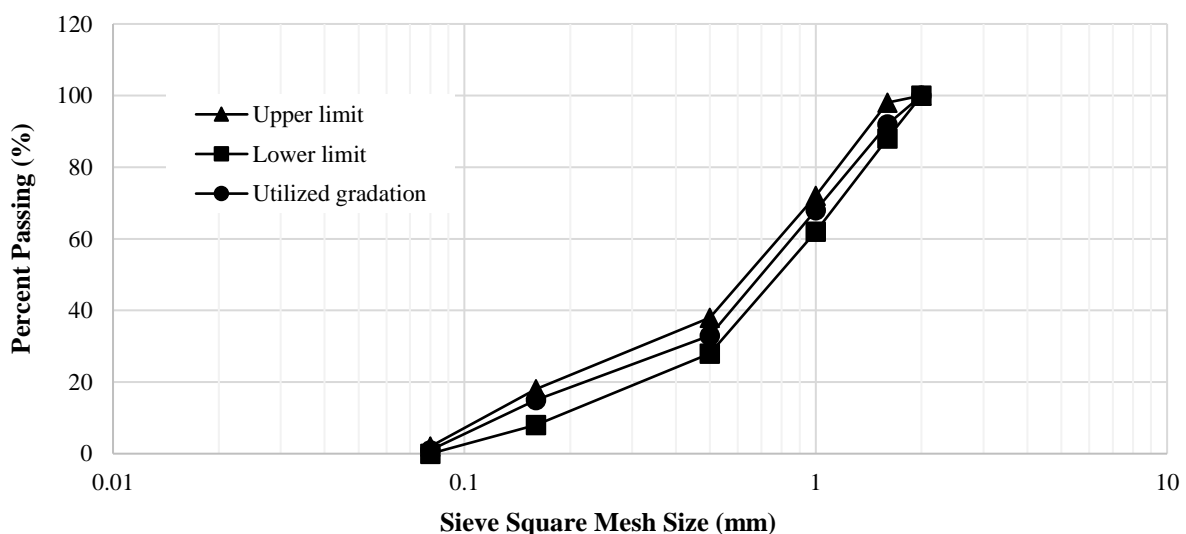
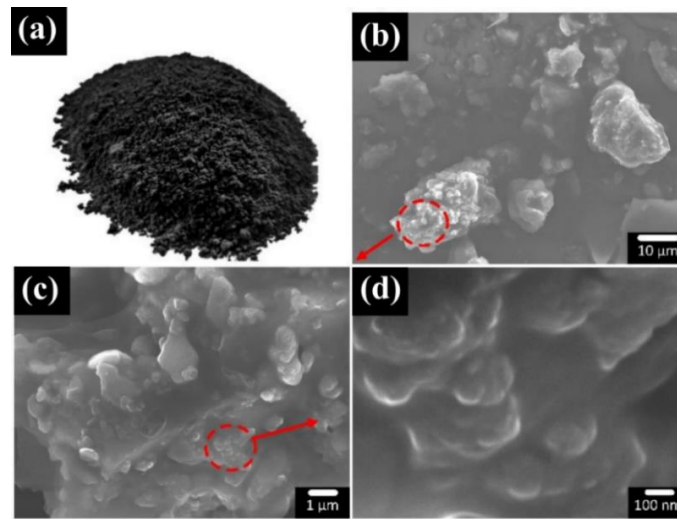


Fig. 1. Silica aggregate gradation based on BS 196-1 (2005)

**Table 1.** The XRF results for the utilized OPC

Chemical compositions (wt%)					
SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	SO <sub>3</sub>
20.74	4.90	3.50	1.20	62.95	3.00

**Fig. 2.** a) The AC powder; and b-d) The SEM image in micro and nano scales**Table 2.** The physical and chemical properties of AC powder

Property or component	AC
Specific gravity	0.92
Particle size (μm)	10 - 70
Specific surface area (m <sup>2</sup> /gr)	4.76
C (%)	74.38
O (%)	17.92
S (%)	7.24

### 3.1. Mix Design

The standard mix design for cement mortar was used as stated in BS 196-1 (2005). According to the British Standard, water-cement and aggregate-cement ratios were 0.5 and 3, respectively. PCE superplasticizer was added to the mix by 0.1% of cement mass. AC powder was replaced with 4, 6 and 8 percent of aggregate. Mix design with no AC content was used as a reference. Mix design for all percentages of replaced AC powder are reflected in Table 3.

### 3.2. Rheology

To evaluate the workability, mini-slump

and flow table tests were carried out according to ASTM C230 (2020). The tests were conducted immediately after mix procedure was complete.

### 3.3. Mechanical Tests

#### 3.3.1. Flexural Strength

Flexural strength was measured by testing six 40 × 40 × 160 mm cubes, moulded for each mix design. Mechanical tests were carried out by a Universal Testing Machine (UTM) based on BS 196-1 (2005). After demolding, specimens were cured in saturated limewater in the room temperature (23 ± 2°C) for 7 and 28 days.

**Table 3.** Mix design of specimens

Mixture	Proportions in Kg/m <sup>3</sup>				
	Cement	Aggregate	Water	AC	superplasticizer
AC 0%	406	1219	203	0	0.41
AC 4%	406	1171	203	48	0.41
AC 6%	406	1123	203	96	0.41
AC 8%	406	1075	203	144	0.41

### 3.3.2. Compressive Strength

To detect the compressive strength of mixtures, portions of flexural prisms were used. Each portion was 40 mm in depth and width and approximately 80 mm in length in accordance with ASTM C349 (2018).

### 3.4. Water Absorption

Investigating the water absorption of samples containing AC powder is one of the integral parts of this study to measure the competency of AC powder in limiting water ingress into concrete. Water absorption rate and volumetric water absorption tests were conducted based on ASTM C1585 (2014) and ASTM C642 (2013), respectively.

In the capillary water absorption rate test, the standard specimens, which are 100 mm diameter discs with a height of 50 mm, were cured for 28 days prior to testing and oven-dried for 24 h at 105 °C and then weighted. It is highly advised to ensure that water is only allowed to penetrate from the specified surface and the flow to be one-directional. To achieve this, waterproof plastic tapes were attached to the surroundings of the samples and a plastic sheet was used on top of it to hinder evaporation. The test started by placing the samples on rods, acting as supports, and immersed in water such that the whole bottom surface and  $2 \pm 1$  mm of the height of them were in contact with water. The mass gain of the specimens was measured at certain time intervals after blotting to remove excess water within 15 s of removal from the pan and then immediately replaced on the support device.

The absorption, as shown in Eq. (1) is defined as the mass of absorbed water per unit area of the surface in contact with water divided by the density of water.

$$I = \frac{m_t}{a \times d} \quad (1)$$

where  $I$ : is the absorption,  $m_t$ : is the change in the specimen weight at the time  $t$ ,  $a$ : is the exposed area of the specimen and  $d$ : is the density of the water.

In addition to the sorptivity test, the volumetric water absorption test was carried out in which the results can indicate the full capacity of the specimen to contain water. Samples were fully immersed in water after being completely dried out. The first weight measurement took place after 48 h of immersion and the test continued by 24 h intervals until the weight alteration was less than 0.5% which implies that the sample is saturated. Mass change percentage with respect to the initial dried mass of the specimen was then reported.

### 3.5. Microstructural Analysis

Fracture surface of cement mortars with and without AC powder were subjected to Energy Dispersive X-ray spectroscopy (EDX) as a means to identify the chemical composition and any cement-carbon reaction in the interfacial transition zone (ITZ).

## 4. Results and Discussion

### 4.1. Flow

The results from the flow table test indicate that the anticipated deterioration in workability can be concerning. However, by the means of the superplasticizer, modification is well possible. The flow feature of the carbon-containing mortars is less than that of the reference mixture yet, considerably acceptable. Both mini-slump and flow table results are rendered in Figure 3. The shape of data indicates a linear relationship between mini-slump results and AC powder content. However, when 8% AC is incorporated in the cement mortar, the flow is so low that special treatments such as addition of superplasticizer are needed. Other types of mixing procedures such as ultrasonication is highly recommended in such situations. Moreover, higher percentages of superplasticizer may not be the best practice due to the fresh plasticity index of the cement matrix and its deteriorating effect on the setting time.

## 4.2. Mechanical Strength

The results of mechanical strength are obtained in 7 and 28 days after moulding. Figure 4 portrays the compressive and flexural strengths. Overall, by replacing 8 percent of aggregate with the AC powder, the compressive strength after 28 days increased nearly 100% of the reference mix design with no AC content. Besides, the flexural strength is either remained the same or barely increased by different AC content. The increase in flexural strength is predominantly due to an increase in the compressive strength and ductility is not expected to change significantly by adding the AC powder. The means and standard deviations for all AC concentration levels are provided in Table 4.

The notable enhancement in the compressive strength can be a function of higher surface area and roughness of AC compared to those of silica aggregate. Therefore, cement can have a better bonding with the AC and as a result, the compressive strength elevates. The AC

powder is also capable of entrapping the water drops (Shahbazi et al., 2020) and cause a decreased practical w/c ratio and consequently, working in favour of strengthening.

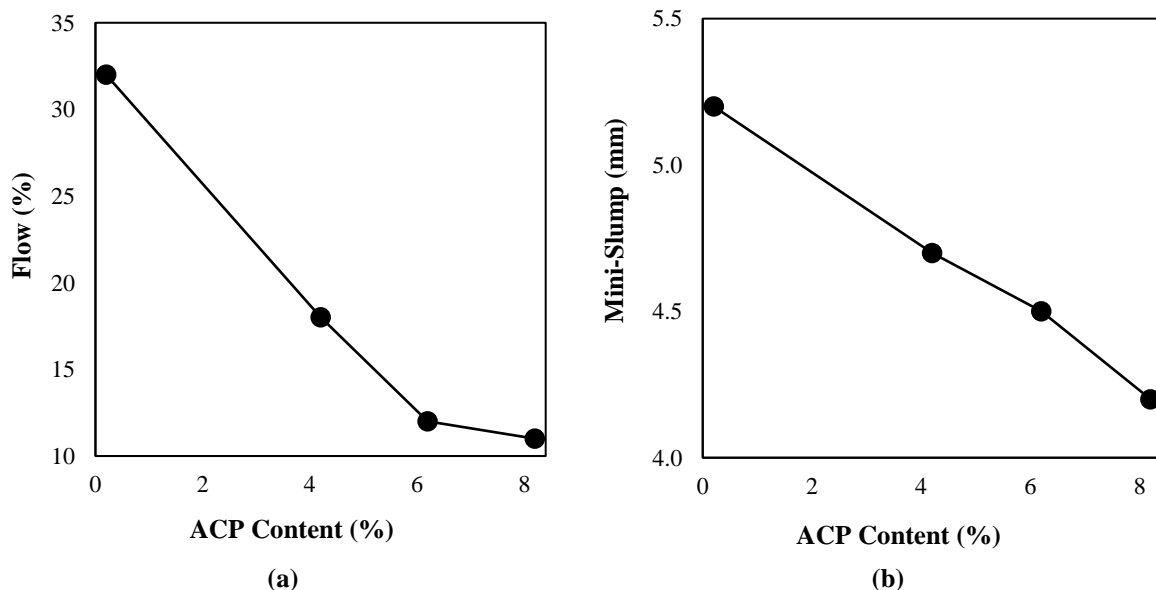
## 4.3. Investigation of Hydrophobicity

One famous way to illustrate the level of hydrophobicity of a surface is to measure the contact angle of a water droplet on that surface (Arabzadeh et al., 2016; Chieng et al., 2018). The greater the internal contact angle is, the more hydrophobic the surface would be. As it can be seen in Figure 5, the water droplet on the control sample was absorbed and spread out far and eventually rested at 31 degrees. While, in the ACP incorporated samples the droplet retained its spherical shape with a contact angle 124, 142, 150 degrees for 4, 6, 8% ACP specimens respectively. The contact angle was measured by image processing technique using ImageJ software (Schneider et al., 2012).

**Table 4.** The mean and standard deviation of mechanical strengths

Mixture	Compressive strength (MPa)		Flexural strength (MPa)	
	7-day cured	28-day cured	7-day cured	28-day cured
ACP 0%	19.3 (1.6)*	24.4 (2.9)	5.4 (0.2)	7.9 (0.2)
ACP 4%	14.6 (1.5)	41.8 (1.6)	5.5 (0.2)	8.6 (0.2)
ACP 6%	17.7 (2.2)	46.5 (2.1)	5.5 (0.3)	8.9 (0.1)
ACP 8%	19.1 (1.9)	50.9 (2.2)	4.8 (0.2)	8.9 (0.2)

\*Standard deviations are presented in brackets.



**Fig. 3.** a) The flow table results; and b) The mini-slump results

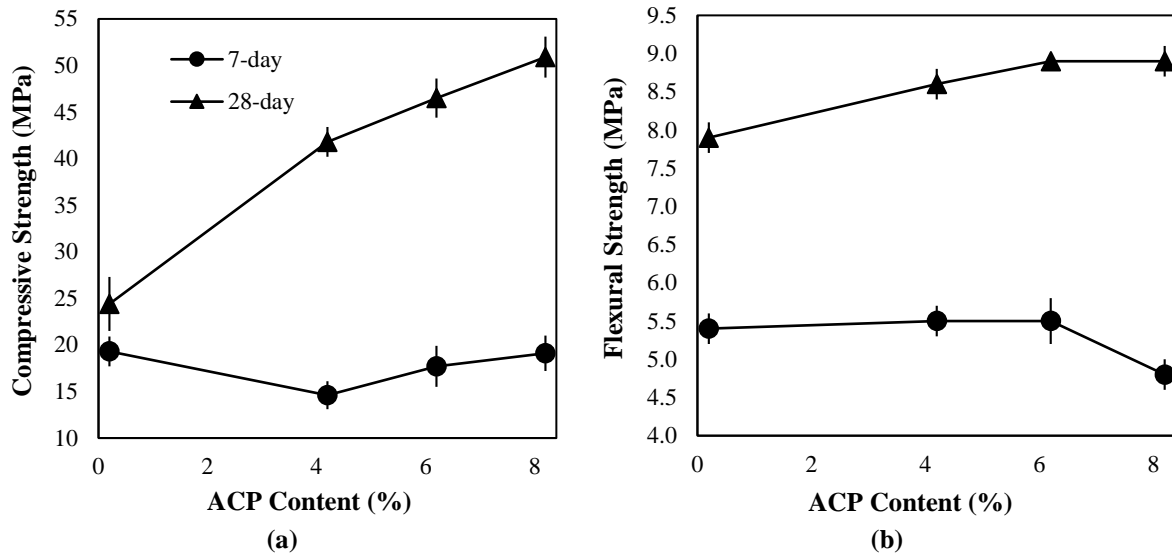


Fig. 4. a) The compressive; and b) flexural strength, of specimens

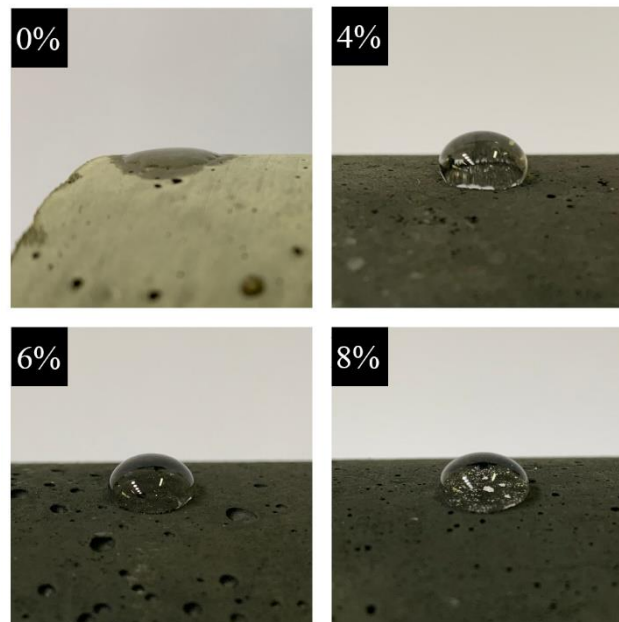


Fig. 5. Effect of AC powder on surface hydrophobicity of cement mortar

#### 4.4. Water Absorption

The absorption rate of the 8 samples including 0%, 4%, 6%, and 8% AC powder, has been plotted versus the square root of time, as shown in Figure 6a. Results illustrate the expected general behaviour in which the rate of absorption, the slope of the graph, decreases as time passes. This was because samples became wetter and the speed of water uptake declined. As can be seen, water ingress has been limited when comparing the control sample to the sample containing 8% of AC powder throughout the whole graph. For instance, the

absorption index of samples containing 4, 6 and 8% ACP, which resembles the mass gain of the sample because of capillary water uptake, has decreased by 14, 17, and 25%, respectively, when comparing to that of the control sample. The results of the volumetric absorption test are depicted in Figure 6b. It is quite clear that AC substantially decreases the total amount of absorbed water dramatically. 54, 56, and 65% reduction in mass gain of the samples with 4, 6, and 8% hydrophobic AC powder was seen.

According to many studies, there is a



direct relationship between water absorbed and the durability parameters such as freeze-thaw cycles (Basheer et al., 2001) and carbonation (Parrott, 1992). Thus, these

results affirm that AC can prepare a hydrophobic medium which may consequently enhance the cement matrix's durability.

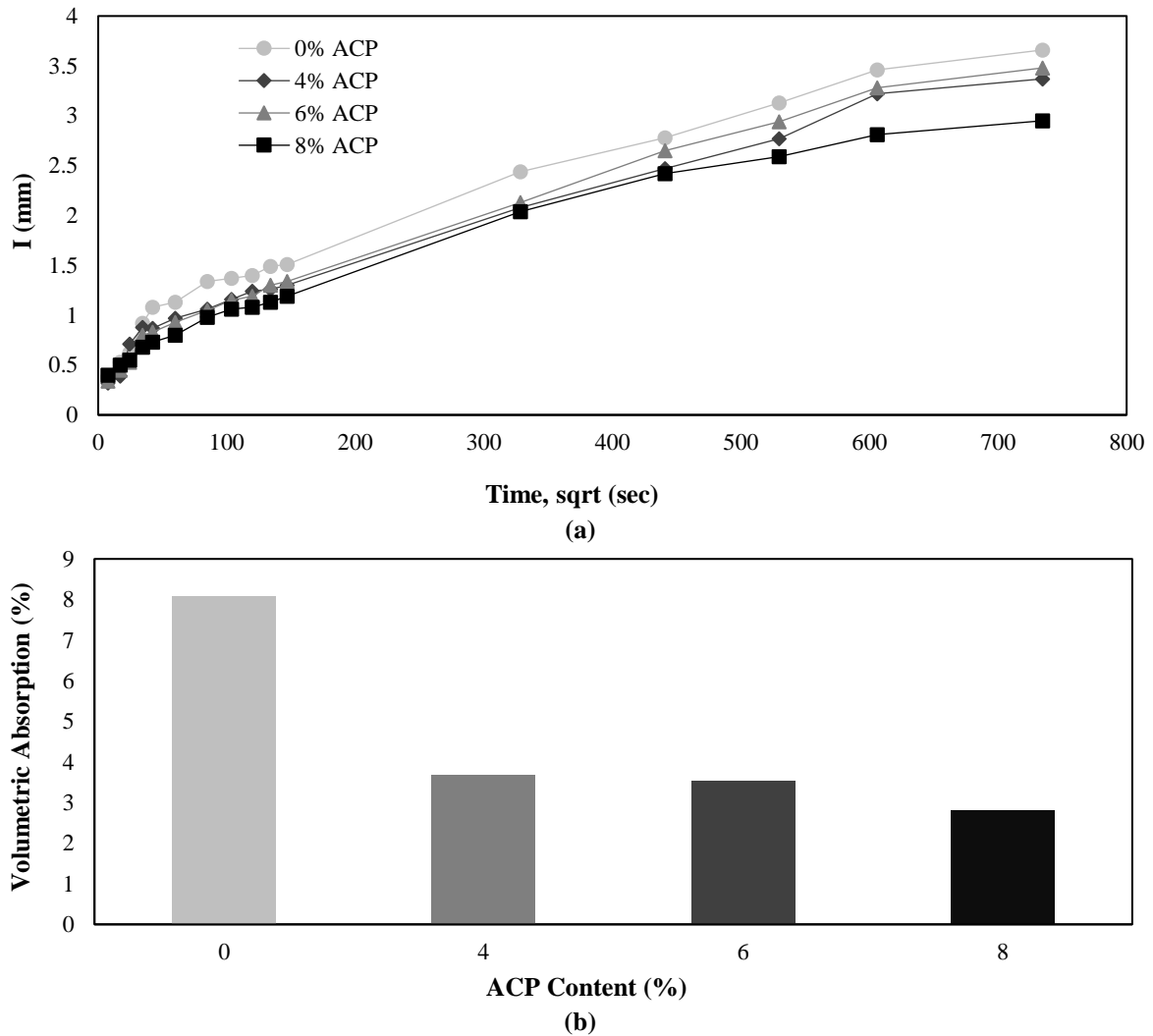


Fig. 6. a) The rate of water absorption; and b) The rate of volumetric absorption

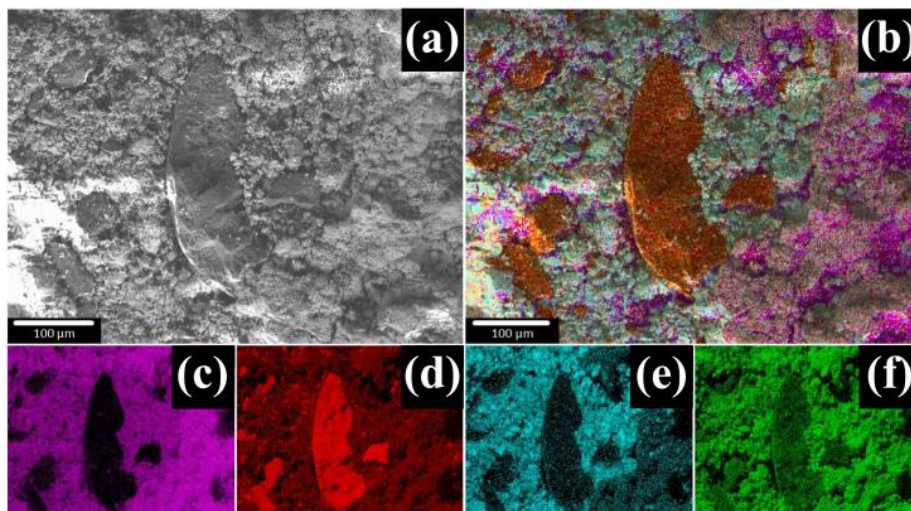


Fig. 7. a-b) SEM and elemental intensity and distribution map of cement mortar with AC; and c-f) Individual intensity and distribution map for predefined elements of calcium, carbon, silicon and oxygen

#### 4.5. Microstructural Analysis

Further investigation on the microstructure of cement mortar with AC powder showed that AC has no chemical reaction with cement when it is used as aggregate. Assuming any chemical reaction, the intensity of calcium must moderately decrease around the carbon particle due to the formation of ITZ by calcium hydroxide molecules. However, it is clear in Figure 7c and 7e that there is no sign of gradual decrease neither for calcium nor silicon intensity near the boundaries of AC powder. This proves the inertia of AC powder in the cement matrix.

#### 5. Conclusions

There is a knowledge gap for recycling AC powder in concrete. This research was done in the first place to enhance the knowledge of mechanical and durability features of concrete with respect to the hydrophobicity of AC. The paper presented the results of the flow table, mini-slump, compressive and flexural strength and water absorption. The following portray the most important findings:

- The inclusion of carbon powder although not affecting the flexural strength remarkably is plausible causation for a sharp increase in 28-day compressive strength.
- ACP 8% mix had the highest increase with a compressive strength of 19.1 and 50.9 MPa in 7 and 28 days, respectively.
- The water absorption rate and total volumetric absorption of the mixtures decreased by increasing the AC content. 8% of AC resulted in a 25 and 65% decline in absorption rate and volumetric absorption, respectively.
- AC powder inclusion resulted in a considerable drop in flow properties. 8% of AC powder was enough to lessen the slump and flow by 88 and 20%, respectively.

The two-way interaction of AC content and compaction energy can be further

investigated by the mean of two-factor factorial design. Furthermore, due to unconformable changes in 7-day and 28-day strengths, the calorimetry tests are essential to analyze the AC effect on the cement hydration. Tests such as sulfate and acid attack, rapid chloride penetration, rapid chloride migration and bulk electrical resistivity can deepen the understandings on exact procedure based on which AC contribute to durability.

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