

1 Article

2 Sustainable Utilization of Corn Stalk Ash and Used Engine Oil 3 as Hydrophobic Admixtures in Cement based composites

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12 Abstract

13 The penetration of water into concrete remains a critical durability concern, as moisture
14 ingress initiates and accelerates a wide range of deterioration mechanisms. At the same
15 time, environmentally harmful waste materials such as cornstalk ash and used engine oil
16 pose significant risks due to air emissions, soil contamination, and water toxicity. This
17 study investigates the potential utilization of corn stalk ash and used engine oil as hy-
18 drophobic admixtures in cement-based composites, thereby offering both performance
19 enhancement and sustainable waste management. Concrete specimens were prepared
20 with corn stalk ash contents of 0%, 2%, 4%, and 8% and used engine oil contents of 0%,
21 0.5%, 1%, 2%, and 4%. Sorptivity, ultrasonic pulse velocity, and visual assessments were
22 conducted to evaluate water ingress and material integrity. Increasing corn stalk ash
23 content generally resulted in higher sorptivity and lower UPV, primarily due to its in-
24 trinsic porous structure. The combined effects of corn stalk ash, used engine oil, and
25 curing age produced nonlinear trends across all measured properties, highlighting the
26 importance of mixture proportions. Overall, the composite containing 2% corn stalk ash
27 and 0.5% used engine oil demonstrated the most effective hydrophobic response, re-
28 ducing moisture penetration while simultaneously diverting waste materials from envi-
29 ronmentally harmful pathways. These findings demonstrate that corn stalk ash and used
30 engine oil can serve as viable sustainable admixtures, contributing to improved concrete
31 durability and reduced environmental impacts.

32 **Keywords:** Hydrophobic concrete, Corn stalk ash, Sorptivity, UPV

34 1. Introduction

35 Concrete remains the most used and essential material in construction industry. The
36 volume of concrete used in construction is nearly twice that of all other building materi-
37 als combined [1]. The importance of concrete is rooted in its desirable properties, in-
38 cluding high strength, durability, moldability, temperature resistance, and low mainte-
39 nance requirements [2].

40 The water penetration has remained a major challenge since the earliest use of con-
41 crete that significantly reduces durability and initiates various deterioration mechanisms,
42 particularly in structures such as basements, reservoirs, and other water-retaining com-
43 ponents. Concrete durability is closely linked to its pore structure, as pores allow the
44 movement of water and dissolved salts. The intrusion of salts through these pores can
45 lead to expansion, cracking, and further degradation, ultimately compromising the
46 long-term performance of the structure [3].

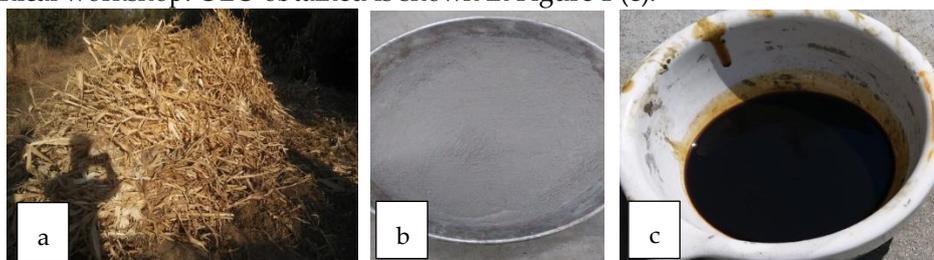
47 Various approaches are used worldwide to enhance the durability of concrete [4-8].
48 One of the techniques is the incorporation of hydrophobic admixtures directly into the
49 concrete mix. These admixtures may include fatty acids, silanes, siloxanes, vegetable oils,
50 or combinations thereof, and have been shown to improve water resistance more effi-
51 ciently [10].

52 In earlier studies, materials such as stillage residue of synthetic fatty acids, oil bi-
53 tumen, fine rubber powder, paper sludge ash, and stearic acid were incorporated into
54 concrete to achieve hydrophobicity; however, many of these additives are costly and not
55 readily available in large quantities [11, 12]. In contrast, agricultural and industrial waste
56 materials such as corn stalk ash (CSA) and waste industrial oil present a more economical
57 and sustainable alternative. CSA, being a fine powder, can act as a filler to increase con-
58 crete density, while waste industrial oil functions as a hydrophobic agent that repels
59 water, reduces porosity, and improves overall durability [13-15].

60 The incorporation of CSA and waste industrial oil as hydrophobic admixtures in
61 concrete presents a promising approach to mitigate durability issues while enhancing the
62 overall quality and reliability of structures. This study investigates the potential of these
63 waste materials to improve water resistance and structural performance, simultaneously
64 promoting sustainable waste management. To assess their effectiveness, sorptivity tests,
65 ultrasonic pulse velocity (UPV) measurements, and visual inspections were performed to
66 evaluate water ingress and material integrity in the modified cement-based composites.

67 2. Materials

68 Ordinary Portland Cement (OPC) Type I, in conformity with the specifications of
69 ASTM C150 was used throughout the experimental work. CSA was obtained from
70 Dhangri village, Mansehra, KPK whose latitude and longitude are 34° 19' 10" and 73° 10'
71 45" respectively. The raw corn stalk procured is shown in Figure 1(a). The collected corn
72 stalks were subjected to uncontrolled open-air burning, and the resulting residue was
73 allowed to cool in ambient conditions. The cooled residue was manually sieved to sepa-
74 rate the fine ash, which was further passed through a #200 sieve Figure 1 (b). The sieved
75 ash was then ground at PCSIR Laboratories, Peshawar, and stored in airtight polyeth-
76 ylene bags for subsequent use. Used engine oil (UEO) was obtained from a local me-
77 chanical workshop. UEO obtained is shown in Figure 1 (c).



78 **Figure 1.** a) Raw Corn stalk; b) Corn Stalk Ash sieved through #200 sieve; and c) Used Engine Oil

80 Chapelle Test reveals that CSA satisfies the minimum requirement set out for poz-
81 zolanic activity i.e. 330 mg/g of Ca(OH)₂/g of pozzolan, irrespective of crystallinity. The

lime consumption shown by the sample is 850.47 mg/g which is 157.72% above minimum requirement. Therefore, it is concluded that CSA is highly chemically active.

Scanning electron microscopy describing the morphological features of CSA are shown in Figure 2. The particle sizes are normally seen to be in the range of $<0.1-70\ \mu\text{m}$. Variety of shapes can be seen and they are designated by the letters A, B and C as shown in Figure 2. The letter A refers to particles having elongated and rough shape. These particles impart interlocking characteristics. The particles/areas designated B refers to very higher surface area of the particles and they can act as filler for pores. Areas designated with letter C shows round/spherical particles. High surface area and porosity may impart water absorption characteristics to CSA. It may also impart adhesive and cohesive properties to CSA

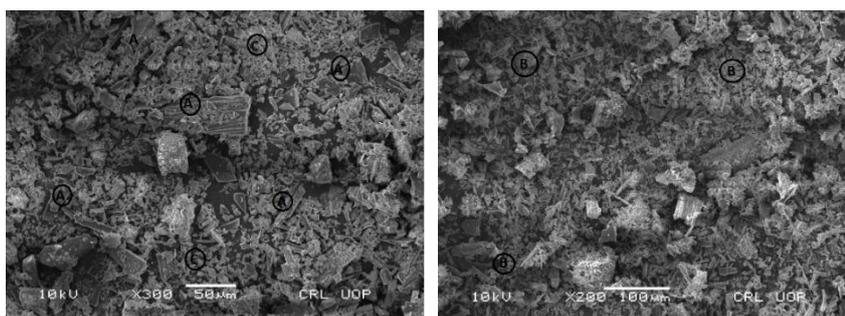


Figure 2. Scanning electron microscopy analysis of Corn stalk ash

Chemical composition of CSA yield by X-ray fluorescence analysis is shown in Table 1. According to ASTM C-608 the sum of silica, alumina and ferric oxides should be at least 70% in a material to fulfill the chemical requirement of pozzolanic material. In the case of CSA, $\text{SiO}_2 + \text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$ is 60.27% which is not confirming to ASTM standards but in accordance with the Brazilian Standard requirement of minimum 50% of these oxides (NBR12653, Materiais pozolânicos. Especificação. Brasil). High amounts of Potassium Oxide (K_2O) and Phosphorus Pentoxide (P_2O_5) were found i.e. 15.97% and 11.42% respectively. This high percentage presence can be attributed to use of different fertilizers. Loss on ignition (LOI) is 2.9% less than maximum specified limit of ASTM i.e. 6%.

Table 1. Chemical composition of Corn stalk ash

Composition	Percentage
Silicon Dioxide (SiO_2)	43.95
Ferric Oxide (Fe_2O_3)	5.61
Aluminum Oxide (Al_2O_3)	10.71
Potassium Oxide (K_2O)	15.97
Calcium Oxide (CaO)	1.04
Magnesium Oxide (MgO)	1.5
Sulfur Trioxide (SO_3)	1.29
Titanium Dioxide (TiO_2)	0.02
Phosphorus Pentoxide (P_2O_5)	11.42
Loss on Ignition	2.9

3. Methods

A total of 120 cement-based composite samples (2" × 2" × 2" cubes) were cast, incorporating CSA and UEO in proportions (x, y)% where x = 0, 0.5, 1, 2, and 4 percent of oil while y = 0, 2, 4, and 8 percent of CSA. Six samples for each proportion were cast to be tested at curing period of 7, 28, and 90 days of curing. The water-to-cement (w/c) ratio was maintained at 0.32, determined through a trial-and-error process. The mix compositions are named as S(x, y).

The sorptivity test was conducted according to ASTM C1585-04 on samples at different ages i.e. 7, 28 and 90 days. Ultrasonic pulse velocity test was conducted on cement composites according to the specifications of ASTM C597-16. This test was conducted on samples at different ages such as 7, 28 and 90 days respectively. The absorption characteristic of cement composites was determined through visual assessment test. In this process a drop of ink was placed on the surface of the samples having different mix proportions. The samples were then visually observed and pictures were taken.

4. Results and Discussion

4.1 Sorptivity Test

The capillary absorption with respect to square root of time for various samples having different combination ratios after 7 days of curing is shown in Figure 3. This procedure was followed for all cement composites samples at age of 7, 28 and 90 days. From analysis of these graphs, sorptivity was worked out by finding slope of the trend line. The sorptivity values were then compared for all samples as shown in Figure 4 at age of 7, 28, and 90 days.

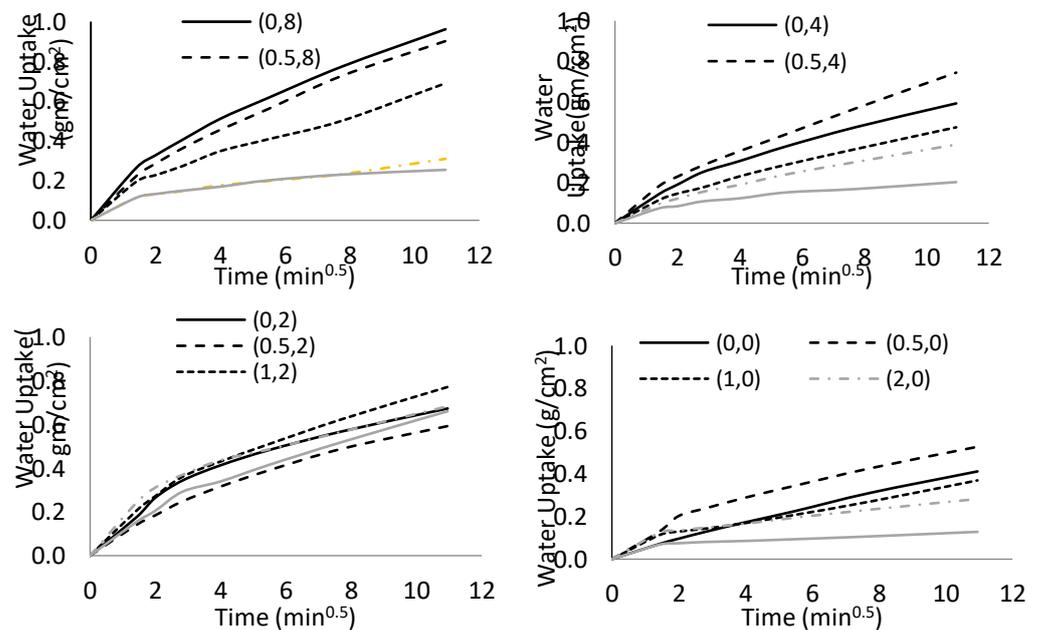


Figure 3. Capillary absorption w.r.t sq. root of time of cement composites after 7 days of curing

The results of 7, 28 and 90 days show almost similar trends as that of percentage water absorption i.e. capillary uptake of water per weight per unit area-square root of time increases with increasing CSA content. The higher porosity and large surface area of CSA resulted in high water absorption due to capillary rise. The maximum sorptivity value is observed for S(0,8) and minimum for S(0.5,0) in 7 days analysis. Also, anomalous behavior can be seen for S(4,4). Analysis on 28 days age show that maximum water uptake is for S(0,8) similar to 7 days but the minimum value has changed and now stands for S(4,0). Anomalous behavior can be seen on 28 days age also for S(1,4) and S(2,8).

The 90 days results represent a clearer picture with trends almost uniform. The maximum water uptake is 0.1 g/cm²-min^{0.5} for S(1,8) which is almost 60% greater than water uptake of control sample i.e. 0.04 g/cm²-min^{0.5}. Similarly, the minimum water uptake was observed for S(4,0) i.e. 0.01 g/cm²-min^{0.5} which is almost 25% less than that of control sample. A decrease in water uptake is observed for S(0.5,2) defying the increasing trend. Similarly, this anomalous behavior can also be seen for S(1,4). This change in trend is due to the combination ratio of CSA and UEO which changes the properties. Also decrease in water uptake was observed with increasing UEO content.

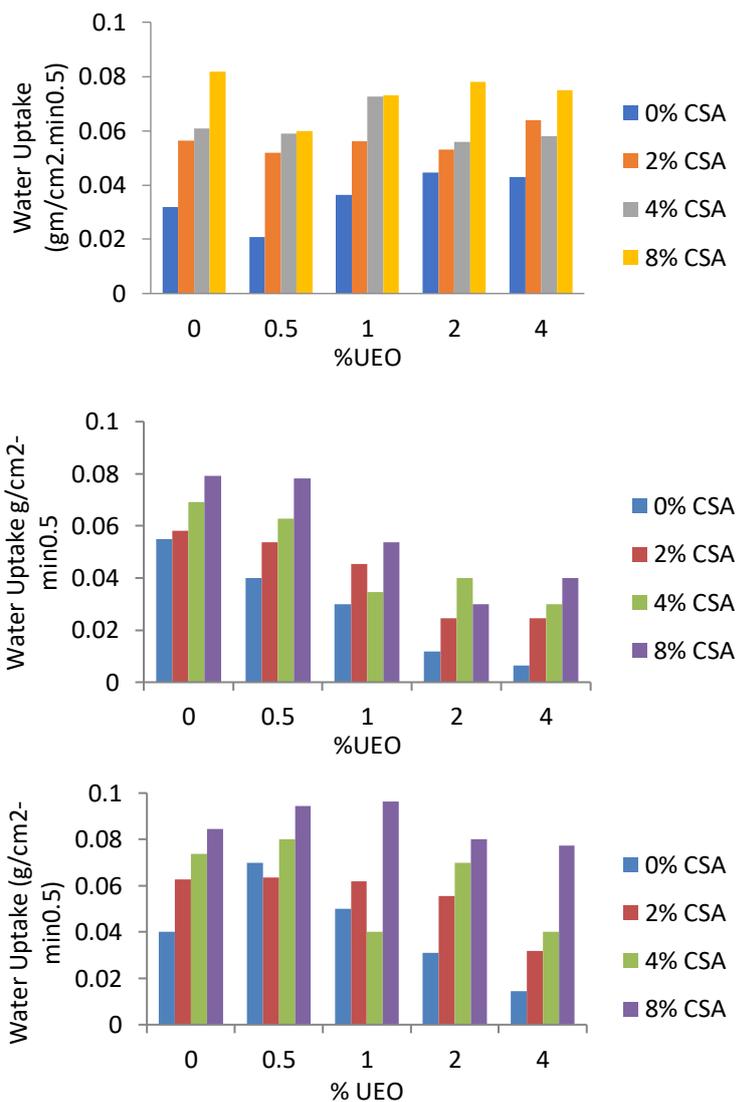


Figure 4. Sorptivity trends of cement composites at a) 7 days; b) 28 days; and c) 90 days

4.2 Ultrasonic Pulse Velocity (UPV)

The results of UPV test conducted at 7-, 28- and 90-days age are shown in Figure 5. The concrete quality grading range in terms of UPV is given in Table 2. UPV is an indirect and non-destructive method of determining the compressive strength. UPV decreased with increasing CSA content, and this is due to the highly porous nature of CSA that left micro pores and capillaries within the concrete mass. Due to this velocity of compressional waves in mixes with greater percentages of CSA is less due to lack of compactness of cement composites.

At 7 days testing the maximum UPV was observed for S(0.5,0) and minimum for S(0,8). Changing behavior was observed for S(4,4). At 28 days, peak UPV was observed for S(2,0) while minimum for S(1,8). The maximum and minimum points have changed with age. Changing behavior is observed for S(1,0). Analysis of 90 days results again shows change in minimum UPV with age. The minimum UPV is for S(0.5,8) having UPV of 2.58 km/sec and falls in the doubtful range of gradation, while maximum is similar to that of 28 days i.e. S(2,0) having value of 3.22 km/sec and falls in the medium range of gradation. Anomalous behavior is observed for S(0.5,2), S(1,4), S(2,4) and S(4,4) i.e. the UPV increases with increasing CSA content.

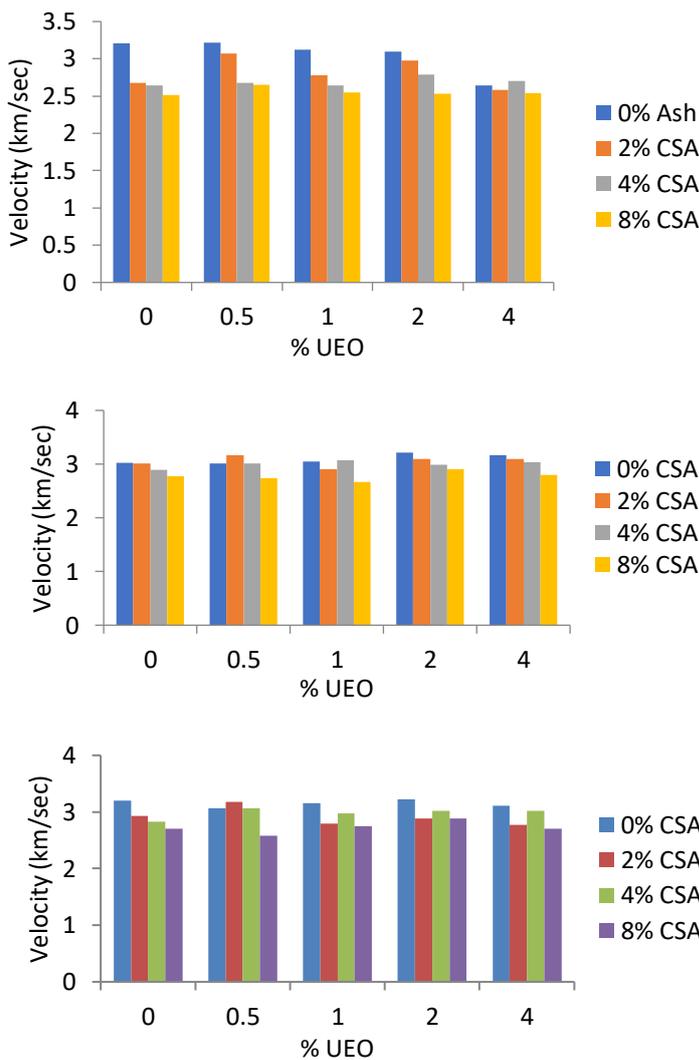


Figure 5. UPV analysis of cement composites at a) 7 days; b) 28 days; and c) 90 days

Table 2. Concrete Quality Grading Range

Concrete Quality Grading as per BIS 13311-92-Part-I	
Pulse Velocity (km/sec)	Concrete Quality Grading
Above 4.5	Excellent
3.5-4.5	Good
3-3.5	Medium
Less than 3	Doubtful

4.3 Visual Assessment Test:

Visual assessment of ink staining on cement composites is shown in Figure 6 for 7, 28 and 90 days curing age. Figure 6a, 6b, and 6c represent control samples i.e. S(0,0) at 7, 28, and 90 days of curing, respectively and corresponding Figure 6d, 6e, and 6f represents admixture sample i.e. S(0.5,2). It can be clearly observed from images of all ages that ink drop on S(0,0) is immediately absorbed and it left a stain on the surface of cement composite. While the ink drops on S(0.5,2) form beads or bubbles on the surface of composite and it can be rolled on the surface. This is due to the hydrophobic effect of the admixture i.e. UEO and CSA on the cement composites. Due to the hydrophobic effect the capacity to absorb the ink drop is reduced by the composites.

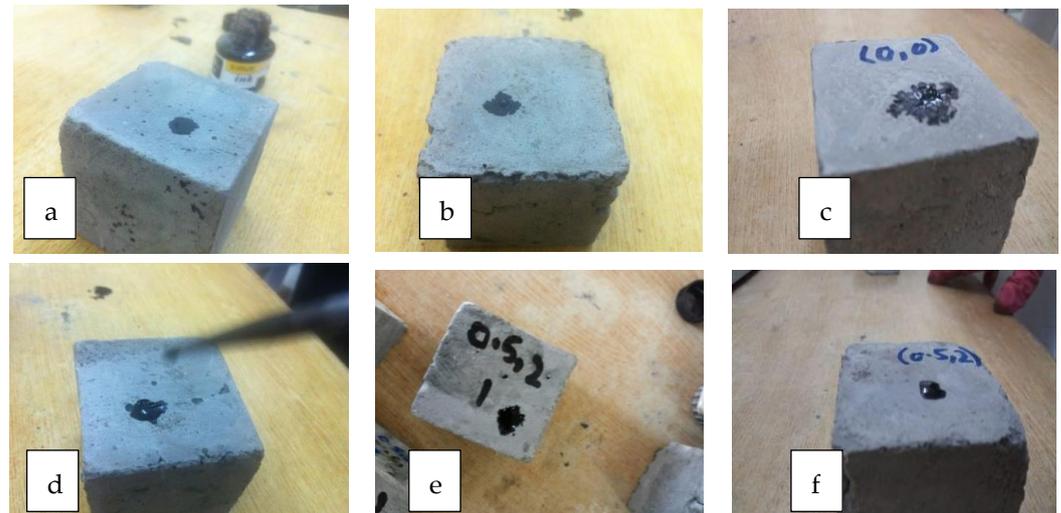


Figure 6. Drop of ink on control sample S(0,0) at curing time of a) 7 days b) 28 days c) 90 days; and on admixture sample S(0.5,2) at curing time of d) 7 days e) 28 days f) 90 days

5. Conclusions

This study examined the combined effects of CSA and UEO on the water penetration behavior and strength of cement-based composites. The results clearly show that CSA alone tends to increase sorptivity and decrease UPV due to its porous and chemically variable structure. However, when CSA is used in combination with small proportions of UEO, a synergistic hydrophobic effect is achieved that significantly reduces moisture ingress. Among all tested cement-based composites, the combination of 2% CSA and 0.5% UEO consistently provided the most favorable performance and is considered as the optimum mix. It provided lower water absorption, improved surface resistance to wetting, and acceptable internal integrity. Moreover, this mixture also aligns with sustainability goals by incorporating two abundant waste materials, thereby reducing their environmental disposal burden. Overall, the findings confirm that CSA and UEO can function as viable hydrophobic admixtures enhancing concrete durability while contributing to waste reduction. Their effects are strongly dependent on dosage, underscoring the need for optimized proportions for practical application.

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