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## Research Article

# **Experimental Investigation on Use of Wheat Straw Ash and Bentonite in Self-Compacting Cementitious System**

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In this research, we evaluated the feasibility of wheat straw ash and bentonite (raw and heated at 150°C for 8 hrs) as secondary raw materials in self-compacting paste (SCP). The fresh and hardened properties of SCP formulations including water and superplasticizer demand, flow behavior, compressive and flexural strength development, water absorption, and acid attack resistance were evaluated. Moreover, porosity, microstructural, and mineralogical investigations were also carried out on SCP formulations. Test results showed that the properties of SCP formulations in fresh state depend on the morphology of secondary raw materials. For heated bentonite and wheat straw ash formulations, the 28 days of compressive and flexural strength were higher or almost similar to reference SCP formulation. Among SCP formulations, wheat straw ash formulation was found to be more effective in consuming free lime and showed significant decrease in porosity with time, which in turn improved the resistance of this SCP formulation against water absorption and acid attack. Based on the test results, it can be concluded that the successful utilization of wheat straw ash and bentonite SCP formulations will offer durable and environmental friendly option to construction industry.

#### 1. Introduction

Self-consolidating concrete (SCC) is by definition a concrete that completely fills the formwork under its own weight while maintaining homogeneity and without any need for consolidation. It deforms under its own weight as a fluid does and holds the particles of all sizes without letting them segregate into ingredients. High deformability and segregation resistance are the two primary properties of SCC. High deformation is achieved by using efficient superplasticizer and high segregation resistance is achieved by using either low water/powder ratio or moderate water/powder ratio with viscosity enhancing agent. Various pozzolanic and non pozzolanic materials such as lime stone powder [1, 2], fly ash [3], silica fume [4], metakaolin [5], rice husk ash [6], and bagasse ash [7], have been used by many researchers in SCC formulation. Some of them are presented here.

Bosiljkov [1] investigated the influence of nonpozzolanic fillers (finely ground limestone and crushed limestone dust) on the fresh and hardened properties of SCC mixes. Test results showed that finer and better-graded limestone dust significantly enhances the deformability of the paste. Moreover, it was found out that with high filler volume, the required SCC properties were achieved at lower water/(cement + filler) ratio. Rizwan and Bier [8] carried out investigation on blends of limestone powder and fly ash on the response of self-compacting mortars (SCM) in fresh and hardened states. It was found that SCM using blended raw materials improved the overall response than those using either of them. The durability of SCC prepared with metakaolin and silica fume in replacement mode was investigated by Hassan et al. [4]. According to the test results, highly durable SCC mixtures were obtained using high metakaolin content with an optimum replacement percentage of around 20. Furthermore, it was found out that the durability of SCC mixtures prepared with higher MK content was superior to SCC containing silica fume.

Several researchers have evaluated the feasibility of using wheat straw ash (WSA) and bentonite in normal concrete [9-11]. However, the data regarding utilization of WSA and bentonite in self-compacting paste (SCP), to the best of our knowledge, is scarce or nonexistent. This research was therefore carried out to evaluate the feasibility of WSA and bentonite self-compacting paste formulations. The SCP formulations were prepared with 10% replacement level. This replacement level was chosen because a smaller amount of secondary raw material (SRM) results in optimum efficiency and gives a large increase in strength while the use of large amount has a smaller effect [12]. The successful utilization in prevailing environment will offer multiple benefits including cost effective solution to construction industry and reduced environmental and health issues and would have positive impact on the durability of the system.

#### 2. Wheat Straw and Bentonite Potential-Global and Pakistan Statistics

Wheat is one of the primary sources of food for 2.45 billion people [13]. In 2014, the annual global production of wheat was around 705 million tons, out of which 75% was produced by 18 countries while around 20% was produced by EU-27 [14]. According to the statistical data [14], Pakistan is ranked at 7 with 3.5% share to the world wheat production. As per Pan and Sano [15], the average yield of straw is around 1.3-1.4 kg per kg of grain. This places the global estimate of wheat straw at around 534 million tons and for Pakistan this value is around 18.46 million tons. The current use of this abundant supply of wheat straw, as described by Zhang et al. [13], is limited to feeding stuff [16], pulp and paper [17], nanomaterials [18], bioethanol [19], fertilizer [20], and construction of mud houses. However, in some parts of the world and especially in underdeveloped and developing countries, wheat straw is burnt in open field causing environmental issues and health problems. Huge amount of ash, obtained after burning of wheat straw, probably has no use and getting rid of it is also a problem. This research therefore evaluates the feasibility of using WSA in SCP formulations.

As far as bentonite is concerned, the total world production of bentonite has increased from 13.3 million tons in 2004 [21] to 15.5 million tons in 2011 [22] while Pakistan's annual production has increased from 6,154 tons in 2004 [21] to 30,840 tons in 2011 [22]. Moreover, Pakistan has millions of tons of bentonite reserves [9]. This research therefore also evaluates the feasibility of using bentonite (raw and heated) in SCP formulations.

#### 3. Experimental Investigation

3.1. Materials and Mix Proportion. Locally available Ordinary Portland Cement (OPC) grade 42.5R in conformity with ASTM C150 [23] was used. Bentonite clay was collected from Jehangira situated at 33°59′56″ latitude and 72°12′47″ longitude according to Survey of Pakistan topographic sheet 43 C/1 [10] and it was studied in raw and heated conditions. Heat treatment was given at 150°C [9] for 8 hrs and the clay was ground according to the procedure mentioned in [10]. The powder was then sieved through number 200 standard mesh and resulting sample was used for SCP formulations. WSA was obtained by burning wheat straw available in 1-5 cm length. The burning of wheat straw was carried out for 5 hrs in a temperature controlled electric furnace having smoke vent at the top. During the entire burning process, temperature was maintained at 670°C as reported to be quite effective to attain good pozzolanic properties [11]. For all SCP formulations, bentonite and WSA were used in replacement mode by 10% weight of cement. A third generation polycarboxylate ester based powder type superplasticizer Melflux 2651 was used to achieve flow level of  $30 \pm$ 1 cm as measured by Hagerman's mini slump. The following specimen designations were used for SCP formulations.

CEM represents reference SCP formulation.

C + 10% WSA represents SCP formulation with 10% wheat straw ash.

C + 10% BN (H) represents SCP formulation with 10% heated bentonite.

C + 10% BN(R) represents SCP formulation with 10% raw bentonite.

*3.2. Mixing Regime.* Hobart Mixer (5 L capacity) was used for mixing SCP formulations. Various mixing regimes [24] are mentioned in the literature; however, after experimenting, the following was adopted. The constituents were manually mixed in dry state for 1-2 minutes followed by feeding into the bowl the dry mixture along with the requisite mixing water predicted by water demand test. A slow mixing (145 rpm) was done for 60 seconds followed by fast mixing (285 rpm) for 120 seconds [25]. The total mixing time was 3 minutes (180 seconds) for all SCP formulations.

3.3. Testing of Specimens. The particle size, surface area, and surface morphology of powdered WSA and bentonite samples were determined by using Laser Granulometer (Coulter LS 230), Brunauer-Emmett-Teller (Finesorb-3010), and Environmental Scanning Electron Microscope (XL 30 ESEM FEG). For chemical composition, X-ray fluorescence (JSX-3100RII) technique was used. The physical properties and chemical composition results of powdered samples are representative of three samples.

The water demand and setting times of reference SCP as well as those containing 10% SRM (in replacement mode) were determined by Vicat apparatus at  $20 \pm 1^{\circ}$ C and  $20 \pm 5\%$  relative humidity. The superplasticizer amount required for a target flow of  $30 \pm 1$  cm was obtained for all formulations

using Hagerman's mini slump cone having dimensions of  $60 \times 70 \times 100 \text{ mm}^3$ . Moreover, the flow behavior of SCP systems was characterized by Hagerman's mini slump cone spread and by V funnel time. The details of the test can be found in [8]. It needs to be pointed out here that the properties of SCP in fresh state represent the average of three specimens.

The flexural and compressive strength tests were carried out as per ASTM C 348 [26] and ASTM C 349 [27], respectively. Flexural strength of SCP formulation is the average of three  $40 \times 40 \times 160 \text{ mm}^3$  specimens while the compressive strength test was done on broken halves after flexural tests and strength represents the average of six specimens. For water absorption, ASTM C 642-04 [28] was followed while for acid attack test procedure mentioned by Rawal [29] was adopted. Three samples each were tested to determine the water absorption and acid attack test.

The porosity, microstructure, and mineralogical phases of SCP formulations were investigated using mercury intrusion porosimetry (MIP), scanning electron microscopy (SEM), and X-ray diffraction (XRD) at the age of 1 and 7 days. For SEM and MIP, two small pieces (approx.  $1 \times 1 \text{ cm}^2$ ) extracted from compression test were immediately immersed in acetone to stop further hydration in the surface zone and cleaned from free particles produced during fracture in an ultrasonic bath. The specimens were then submerged in isopropanol to remove traces of acetone left in the small pieces [30]. For SEM observations, the specimens were further treated for preparations normally performed including vacuum drying and gold coating while for MIP the samples were dried in oven at 105°C for 24 hrs before testing. Moreover, for MIP, the contact angle of 140° was selected. For XRD, two to three small pieces obtained from compressive tests were ground and the powdered samples were used for obtaining XRD diffractograms in the 5–70°  $2\theta$  range.

#### 4. Results and Discussions

#### 4.1. Characterization of Powdered Samples

4.1.1. Physical Properties and Chemical Composition of Powders. The physical properties and chemical composition of powders are shown in Table 1. The average particle size of bentonite is around one-fifth of the average cement particle size. Thus, it has comparatively large surface area. It can be seen that heat treatment is effective in reducing the moisture content of bentonite. It is believed that the loss of moisture might have increased the internal porosity of bentonite and thus resulted in increased surface area. Metha and Monteiro [31] described that heat treatment can improve the reactivity of natural pozzolans in cement paste and resultantly enhance certain properties of concrete. It is worth mentioning here that both the raw and heated bentonite meet the requirements of chemical composition of natural pozzolan as laid down in ASTM C 618-01 [32].

The chemical composition of WSA shows  $SiO_2$  and  $K_2O$  as major constituents. According to Biricik et al. [11, 33], the chemical composition of WSA depends upon number of factors such as type of soil for growing wheat plant, the type of fertilizers used, environment, burning temperature,

burning volume, and duration of burning. Visvesvaraya [34] pointed out that  $SiO_2$  content in WSA is dependent on soil and environmental conditions existed during development of wheat crop.

4.1.2. Surface Morphology by Scanning Electron Microscopy (SEM) and Mineralogical Composition by XRD Analysis. The size, shape, and texture of SRMs are important parameters to understand the response of SCP formulations. The micrographs of bentonite (raw and heated) and WSA are presented in Figure 1. Bentonite particles show layered structure which attracts and binds water molecules to its vast inner and outer surface area. The layered structure would also require a higher dosage of superplasticizer to overcome internal friction during flow. It can be seen that particles of raw bentonite show agglomeration due to the presence of internal moisture, whereas heated bentonite particles, in general, are separated. It can also be seen that the average particle size of bentonite clay ranges from 3 to  $5\,\mu$ m, which confirms the value given in Table 1 by laser technique. As far as the micrographs of WSA are concerned, the shape of the particles varies from angular to flat and elongated while the texture appears to be rough and abrasive. The micrograph also shows that the particles contain surface pits. All these, in turn, would increase the water demand of the SCP system to attain consistency.

The XRD of bentonite (raw and heated) and WSA is given in Figure 2. The heated and raw bentonites are composed of quartz, illite, montmorillonite, and clinochlore. Moreover, the heated bentonite seems to be relatively less crystalline than the corresponding raw form. As far as WSA sample is concerned, it possesses a large amorphous portion accompanied by minor traces of quartz and cristobalite. Furthermore, traces of kyanite were also found.

#### 4.2. Fresh and Hardened Properties of SCP Formulations

4.2.1. Water Demand, Superplasticizer Demand, and Setting Time. The water and superplasticizer demands as well as setting times of SCP formulations for a target flow of  $30 \pm 1 \text{ cm}$  [25] are presented in Table 2. In comparison to CEM, both bentonite and WSA SCP formulations showed higher water and superplasticizer demand. For bentonite, the higher water and superplasticizer demand are due to the larger surface area arising from smaller particle size and layered structure which attracts and binds water molecules. In addition, in comparison to raw bentonite, heated bentonite showed higher water and superplasticizer demand. During thermal treatment, internal moisture from bentonite was extracted resulting in larger surface area and hence increased water and superplasticizer demand. Also, the results reported are in line with the available literature on bentonite used in normal cement concrete [9, 35]. As far as WSA is concerned, the increase in water demand with respect to CEM is due to particle shape (angular to flat and elongated), texture (rough and abrasive), and larger surface area. For autoclaved normal cement mortar prepared with WSA, Al-Akhras and Abu-Alfoul [36] reported decrease in flow table values with increasing WSA replacement by weight of cement. Thus,

Parameters	Cement (42.5R)	WSA	Bentonite (H)	Bentonite (R)	
	Phy	sical properties			
Mean particle size ( $\mu$ m)	22.00	12.30	4.32	4.75	
Surface area (cm <sup>2</sup> /g)	1,650	3,200	4,800	2,535	
Specific gravity	3.10	2.30	2.79	2.82	
Moisture content (%)	2.90	_	0.05	3.20	
	Chen	nical analysis (%)			
SiO <sub>2</sub>	17.15	73.95	53.96	49.38	
TiO <sub>2</sub>	0.32	1.92	0.93	1.85	
$Al_2O_3$	5.60	0.91	20.27	17.54	
Fe <sub>2</sub> O <sub>3</sub>	3.21	1.15	8.92	10.24	
MgO	1.44	1.83	4.02	3.80	
CaO	64.09	5.21	7.05	9.54	
K <sub>2</sub> O	1.19	11.51	3.93	4.83	
	Strength	activity index (SAI)			
*7 days	100	96	94	77.26	
*28 days	100	104	99	85.18	

TABLE 1: Physical and chemical properties of powders.

\* SAI ≥75% of CEM (ASTM C618).

suggesting increase in the water demand of WSA mixes. It will be pertinent to mention here that SCM formulations using fly ash require less water and superplasticizer demand for a specific target flow. The lesser water and superplasticizer demand of fly ash formulations are due to their spherical shaped particles, which during flow roll over one another, reducing the internal friction [37, 38]. However, for specific target flow, SCM/SCC formulations prepared with limestone powder (LSP) and metakaolin (MK) require more water and superplasticizer [4, 5, 8, 25]. The higher demand for LSP is due to their irregular, rough, and patchy particle characteristics while for MK, the layered structure provides sites for internal friction during flow. The results of setting time presented in Table 2 show increase in the setting time of SCP formulations with addition of WSA and bentonite. The increase in the setting time of SCP formulations varies according to the individual characteristics of each SRM used.

4.2.2. Flow. After establishing the water demand, superplasticizer demand, and the setting times, the next parameter investigated for SCP formulations was flowability. The results of Hagerman's mini slump cone time for a spread of 25 cm (T25 cm) and V funnel time of various formulations are shown in Figure 3. In T25 cm cone flow time, mostly internal friction has to be overcome during unconfined flow of selfcompacting paste while in V funnel time, both internal and external frictions (between self-compacting paste and gradually reducing funnel section) are present which increase the overall friction and thus the flow time [8]. Increase in T25 cm and V funnel time of SCP formulations containing bentonite and WSA indicate higher friction offered during flow. For the case of bentonite, friction was offered by the layered structure while for WSA, it is angular to flat and elongated shape, rough and abrasive texture and small surface pits were possible sites providing friction. In addition, the presence of internal free moisture in raw bentonite and its

comparatively lesser surface area than "heated" bentonite (Table 1) indicates that more effective water is available in the system to help the flow. This makes the former SCP formulation less viscous than the latter. In summary, the flow of SCP formulation depends heavily on surface morphology and its internal porosity in addition to other influencing factors like mixing regime, sequence of admixtures addition, and water/superplasticizer contents.

4.2.3. Strength Activity Index (SAI). As per ASTM C618, SAI at 7 and 28 days should be 75% of the reference cement mix. SAI results presented in Table 1 show that all the specimens meet the requirements of ASTM C618 [32]. The mixes show higher rate of reactivity at 28 days than at 7 days suggesting increase in the reactivity rate. It can thus be inferred that OPC hydration and pozzolanic reaction have contributed to higher strength development rate of the SCP formulations. Mirza et al. [9] found out that Karak bentonite heated at 150°C for 3 hrs had higher SAI than control and all other heated bentonites (250°C, 500°C, 750°C, and 950°C).

4.2.4. Compressive and Flexural Strength. The compressive and flexural strength results are shown in Figure 4. Up to the age of 7 days, the CEM SCP formulation showed higher compressive and flexural strength than SCP formulations containing WSA and bentonite. According to the available literature [31, 39], the strength gain in mixes containing pozzolan is generally slow at early ages. However, at 28 days, the SCP formulations with WSA and heated bentonite showed higher or almost similar compressive and flexural strength than CEM. The increase in strength can be attributed to pozzolanic reaction that takes place at a slower rate than the hydration of cement, better pore refinement effect, filler effect, and SRM's particle characterization. It is reported [8] that margin of strength increase is dependent upon the degree of pozzolanic activity of the SRM used.





FIGURE 1: Micrographs of (a, b) raw bentonite; (c, d) heated bentonite; and (e, f) WSA samples.

TABLE 2: Water	demand, super	plasticizer o	demand, and	setting time	of SCP f	ormulations.
				····		

Formulation	Water demand (mass% of cement)	Superplasticizer demand (mass% of cement)	Setting time (min)		
	water demand (mass/o or cement)	Superplasticizer demand (mass/) of cement/	Initial	Final	
CEM	28	0.146	225	310	
C + 10% WSA	36	0.260	270	365	
C + 10% BN (H)	33	0.320	260	360	
C + 10% BN (R)	32	0.270	265	370	

4.2.5. *Water Absorption.* Water absorption results are presented in Figure 5. In comparison to CEM, SCP formulations containing WSA and bentonite showed decreased water absorption. It is due to the fact that the reaction between these pozzolans and calcium hydroxide from hydrated cement paste is lime consuming. In addition, it would be shown later that these pozzolans are effective in reducing the porosity of the system which, in turn, improves the durability of the system.

4.2.6. Resistance to Acid Attack. Resistance against acid attack of SCP formulations, with and without SRM, was measured in terms of percentage reduction in weight and strength after immersing the samples in 5 percent hydrochloric acid and



- $$\begin{split} & \text{C: cristobalite; SiO}_2, \text{syn.; tetragonal} \\ & \text{C! clinochlore; } Mg_{3,75}Fe_{1,25}^{2,2}Si_3Al_2O_{10}(OH)_8, \text{syn.; monocline} \\ & \text{I: illite; } K_{0.6}(H_3O)_{0.4}Al_{1.3}Mg_{0.3}Fe_{0.1}^{2+}Si_{3.5}O_{10}(OH)_2\cdot(H_2O), \\ & \text{syn.; monocline} \\ & \text{K: kyanite; } Al_2SiO_5, \text{syn.; triclinic} \\ & \text{Q: quartz; } SiO_2, \text{syn.; hexagonal} \\ & \text{M: montmorillonite; } Na_{0.2}Ca_{0.1}Al_2Si_4O_{10}(OH)_2(H_2O)_{10}, \end{split}$$
- syn.; monocline

FIGURE 2: XRD of powdered WSA and bentonite (heated and raw) samples.



FIGURE 3: Flowability (T25 cm time and V funnel time) of SCP formulations.

5 percent sulfuric acid solutions for 14 and 28 days. The results are presented in Figure 6. The weight loss observed in the SCP formulations was due to the loss of cohesiveness of the cement hydration products [31]. It can be seen that the amount of weight loss was maximum for CEM in both of the acid solutions. The poor performance of CEM against acid attack is due to the fact that it releases considerable portion of free calcium hydroxide that reacts with the acid and a soft and mushy mass is left behind. For SCP formulations containing pozzolans, the free calcium hydroxide reacts with silica present in the mix to form secondary silica gel and thus reduces the amount of free calcium hydroxide. This, in turn, made the SCP formulations more durable. In addition, the lower loss in WSA mix was due to the relatively high content



FIGURE 4: Variation in (a) compressive strength; (b) flexure strength of SCP formulations.



FIGURE 5: Water absorption of SCP formulations at 28 days.

of silica present in the mix for reaction with free calcium hydroxide and thereby making it more durable. It is worth mentioning that the amount of secondary C-S-H gel and subsequent reduction in free calcium hydroxide is dependent on the percentage of pozzolan in the mix. It can also be seen that more deterioration was caused by sulfuric acid than by hydrochloric acid. It is due to the fact that in case of sulfuric acid, a product called calcium sulfoaluminate hydrate also known as ettringite  $[Ca_6Al_2(SO_4)_3(OH)_{12}.26H_2O]$  is formed, which expands and hence causes disruption of the set cement



FIGURE 6: Effect of (a) HCl; (b)  $H_2SO_4$  on the weight loss of SCP formulations.



FIGURE 7: Effect of (a) HCl; (b) H<sub>2</sub>SO<sub>4</sub> on the strength loss of SCP formulations at 28 days.

paste [31], whereas no such product is formed in case of hydrochloric acid.

The results of compressive and flexure strength after exposure to HCl and  $H_2SO_4$  are presented in Figure 7. CEM SCP formulation showed maximum strength loss. For HCl immersion, the loss in compressive and flexure strength was up to 21 and 28% while for  $H_2SO_4$  immersion, the loss was up to 41 and 44%, respectively. It can also be seen that WSA SCP formulation showed better performance. It would be shown later that the WSA SCP formulation was more effective in consuming free lime. Moreover, the improved performance of WSA SCP formulation was due to lowest porosity and average pore radius as determined by MIP. Based on the test results, it can be deduced that these SCP formulations can be used in acidic environment such as chemical industries, sewer pipes, foundations in ground waters containing high concentration of sulfates, and near sea water.

4.2.7. Scanning Electron Microscopy and Energy Dispersive X-Ray Spectroscopy of SCP Formulations. The micrographs of SCP formulations at the age of 1 and 7 days along with oxide composition extracted from EDX are displayed in Figure 8 and Table 3. All formulations showed the growth of ettringite in the form of needle-shaped crystals, hexagonal-prismatic crystals of calcium hydroxide and calcium silicate hydrate (C-S-H). The oxide analysis of cement paste prepared with WSA and bentonite showed reduction in the content of CaO, thus testifying the efficiency of these pozzolans in reducing calcium hydroxide and transforming it into secondary C-S-H gel. It can also be seen that WSA is more effective in consuming CaO and thereby producing SCP formulation with improved mechanical properties and durable system. However, it should be noted here that for cementitious systems, the analysis appeared to be of just qualitative nature and definite quantitative results should not be expected due to large point to point variation in cement based materials.

4.2.8. Mercury Intrusion Porosimetry (MIP) of SCP Formulations. The porosity and pore size distribution were measured using MIP. The results are reported in Table 4. The results clearly indicate decrease in porosity and average pore radius with age. A significant decrease in porosity and average



FIGURE 8: Micrographs of hydration products along with EDAX of (a, b) CEM; (c, d) C + 10% WSA; (e, f) C + 10% BN(H); and (g, h) C + 10% BN(R) at 1 and 7 days.

pore radius was observed by the addition of pozzolans as compared to reference SCP formulation. This is one of the reasons contributing to better mechanical and durability performance of these pozzolans SCP formulations. Among SCP formulations containing pozzolan, WSA formulation showed lowest porosity and average pore radius at the age of 7 days. This explains why WSA SCP formulation showed better mechanical and durability performance among all other SCP formulations.

4.3. XRD Analysis of SCP Formulations. The mineralogical analysis by XRD in the  $2\theta$  range from 5 to 70° was carried out to detect changes in the hydration products due to the presence of WSA, raw and heated bentonites at the age of

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FIGURE 9: XRD of SCP formulations at (a) 1 day (b) 7 days.

TABLE 3: Variation in oxide composition of SCP formulations at 1 and 7 day	.ys
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Oxides	С	CEM		C + 10% WSA		C + 10% BN (H)		C + 10% BN (R)	
	1 day	7 days	1 day	7 days	1 day	7 days	1 day	7 days	
CaO	55.84	59.35	58.32	49.59	54.29	48.06	55.24	52.18	
SiO <sub>2</sub>	23.16	8.78	25.32	8.87	29.24	9.50	25.24	9.80	
SO <sub>3</sub>	7.34	17.44	6.75	23.30	8.49	19.75	7.49	22.15	
$Al_2O_3$	7.92	13.43	7.29	14.12	7.59	15.25	6.83	14.45	
Fe <sub>2</sub> O <sub>3</sub>	1.28	3.57	4.52	1.59	2.80	1.55	4.40	1.69	
K <sub>2</sub> O	1.20	1.06	0.68	0.71	1.32	1.22	1.08	1.05	
MgO	2.48	0.40	1.19	0.56	1.29	0.52	2.10	0.62	

TABLE 4: Pore sizes and total porosity of SCP formulations at 1 and 7 days.

Sample ID	Avg. pore radius (nm)		Max. pore radius (nm)		Total porosity (%)		Porosity (%) (diameter ≤100 nm)		Porosity (%) (diameter ≥100 nm)	
	1 d*	7 d	1 d	7 d	1 d	7 d	1 d	7 d	1 d	7 d
CEM	18.5	10.2	56728.5	57355.7	15.3	16.6	45.4	68.2	54.6	31.8
C + 10% WSA	19.4	5.7	57011.4	52945.6	17.5	12.5	43.7	84.5	56.3	15.5
C + 10% BN (R)	20.2	7.1	55572.5	56169.2	20.1	13.5	44.3	81.3	55.7	18.7
C + 10% BN (H)	19.7	6.5	55653.4	54521.6	18.4	12.6	42.9	78.4	57.1	21.6

\* "d" denotes number of days.

1 and 7 days. The XRD patterns of hydrated cement paste prepared with and without SRM are shown in Figure 9. The reduction in the diffraction peaks of calcium hydroxide in WSA and heated bentonite SCP formulations indicates the efficiency of these materials as pozzolan. The reduction of calcium hydroxide in these mixes is also responsible for improved compressive and flexural strength and better resistance against water absorption and acid attack. In addition, all SCP formulations showed the presence of ettringite  $[Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26(H_2O)]$ , hatrurite  $[Ca_3SiO_5]$ , and larnite  $[Ca_2(SiO_4)]$  at 1 and 7 days.

#### 5. Conclusions and Recommendations

This research work evaluated the effect of WSA and bentonite (raw and heated) on the overall response of SCP formulations. The characterization of SRMs is essential as they affect water demand, superplasticizer demand, flow behavior, strength, durability, microstructure, and mineralogy of SCP formulations. Based on experimental investigations, the following conclusions can be drawn.

- (i) Bentonite and WSA SCP formulations showed higher water and superplasticizer demands. For bentonite, the increase in demand is due to larger surface area arising from smaller particle size and layered structure while for WSA the increase is due to particle shape, texture, and larger surface area. In addition, due to individual characteristics of each SRM, the SCP formulations showed increase in flow time indicating high friction offered during flow.
- (ii) The pozzolanic SCP formulations showed better performance than reference SCP formulation due to reaction of these pozzolans with free lime, which enhances the mechanical and durability performance by reducing free lime content.
- (iii) Among all SCP formulations, WSA formulation showed the best mechanical and durability performance due to better free lime consuming ability and reduced porosity of the system. Moreover, in comparison to raw bentonite, heat treated bentonite at 150°C improved the mechanical and durability performance of SCP formulations.
- (iv) The utilization of WSA and bentonite in SCP formulations provides cost effective, durable, and environmental friendly option to construction industry.

It is suggested that further research should be carried out to evaluate the long-term performance of these SRMs with different replacement levels in self-compacting paste system. The long-term performance includes strength, durability, microstructural, and mineralogical analyses. In addition, detailed studies should be carried out on self-compacting mortar and concrete prepared with these SRMs.

#### Abbreviations

- BN: Bentonite
- CEM: Reference self-compacting paste formulation
- EDX: Energy dispersive X-ray spectroscopy
- LSP: Lime stone powder
- MK: Metakaolin
- MIP: Mercury intrusion porosimetry
- OPC: Ordinary Portland Cement
- SCP: Self-compacting paste
- SCM: Self-compacting mortar
- SCC: Self-consolidating concrete
- SAI: Strength activity index
- SEM: Scanning electron microscopy
- SRMs: Secondary raw materials
- WSA: Wheat straw ash
- XRD: X-ray diffraction.

#### **Conflict of Interests**

The authors declare that there is no conflict of interests regarding the publication of this paper.

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