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# Efficiency of waste marble powder in controlling alkali–silica reaction of concrete: A sustainable approach



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#### HIGHLIGHTS

• Influence of WMP in controlling the ASR expansion was examined.

• Micro-structural behavior due to ASR was explored using SEM and EDS analysis.

• WMP can be effectively used to control ASR expansion leading to sustainable and environment friendly construction.

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#### ABSTRACT

Recycling of wastes in building materials is gaining a lot of attention worldwide. This not only conserves natural resources but also enhances the properties of existing building materials leading to economical and environment friendly construction. The main aim of this study was to explore the efficiency of waste marble powder (WMP) in controlling alkali silica reactivity (ASR) of concrete. For this purpose, WMP was obtained from a marble industry. To initiate the ASR phenomena, reactive aggregate was used in the study. Mortar bar specimens prepared with WMP as cement replacement material at 10%, 20%, 30% and 40% replacement levels (by cement weight) were evaluated in the standard ASTM C1260 test method. Compressive strength and thermal analysis tests were performed to investigate the effect of WMP on strength development of concrete. Results of compressive strength and thermal analysis showed improved strength after 10% of cement replacement with WMP. Moreover, 28% and 50% reduction in mortar bar expansion was observed after replacing 10% and 40% of cement with WMP, respectively. Scanning electron microscopic images also showed no signs of ASR cracking for mortar bars incorporating WMP. However, presence of cracks due to ASR was observed in control specimens. Furthermore, energy disperse X-ray spectroscopy (EDS) showed that amount of alkalies reduced after replacing cement with WMP, leading to overcome ASR expansion. Therefore, based on the results WMP can be effectively used to control ASR expansion leading to durable, sustainable and economical construction.

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#### 1. Introduction

Problem of alkali silica reaction (ASR) in concrete is common around the world. Alkalies (mainly due to cement) release hydroxyl ions in concrete. Hydroxyl ions have the tendency to react with silica of reactive siliceous aggregates. Amount of hydroxyl ions increases as a result of higher alkalies leading to rapid reaction [1]. This reaction results into the formation of ASR gel, which absorbs moisture/water and expands. ASR gel can cause minor cracking leading to major structural distress. It is a time taking process and results into damage of concrete structures.

ASR is considered deadly for concrete and referred as "cancer of concrete" [2]. Different factors like, reactive aggregates, alkali content and available moisture play an important role in ASR propagation in concrete. Mechanical properties as well as durability properties are greatly affected as a result of ASR in concrete [3,4]. Different case studies are available regarding ASR in structures [5]. Problem of ASR was reported in different structures such as hydroelectric plant in Atlantic Canada, Elgeseter Bridge Norway, Warsak and Tarbela dam in Pakistan [6–8]. Tensile strength along with elastic modulus are the properties of concrete, considered greatly affected by ASR [3,9].

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A lot of researches deal with the ways to control ASR [1]. Use of waste materials is considered as the most suitable way to control ASR [10]. Alkalies concentration in concrete can be reduced by using waste materials like silica fume, low calcium fly ash, slag and other waste materials leading to overcome ASR problem [10]. Effectiveness of waste materials against ASR is mainly due to reduced porosity, alkalies reduction and decreased aggregates silica dissolution [11].

Marble has been used in construction works for a long time [12]. It is mainly used in tiles and indoor flooring [13]. Marble deposits are present in many parts of the world including Italy, Brazil, Spain, Sweden, Belgium, France and Egypt [14,15]. In Turkey only, 40% of world's marble deposits are present [16]. A lot of marble is wasted every year as a result of guarrying operations and processing works. Wastage of marble only during processing and polishing work is approximately 70% of the total marble waste generated [13]. This waste is dumped in vacant spaces or discharged into the water bodies leading to serious environmental issues [17]. Now a days, researchers are trying to utilize waste materials in construction to control environment related issues [18-21]. Utilization of waste marble powder (WMP) in construction activities is gaining a lot of attention. WMP can be used as a potential stabilizing agent to improve the properties of weak clayey soils [12]. WMP (up to 10%) in replacement of cement can be effectively used in concrete [22,23]. According to Aliabdo et al. and Corinaldesi et al. [24,25], filler effect of WMP in concrete resulted into improved properties. Utilization of WPM in the replacement of cement has positive effects on durability properties [26]. Dense concrete can be prepared using WMP leading to improved resistance to chloride migration, permeation and corrosion [27]. Furthermore, economical and eco-friendly concrete can be produced by utilizing WMP [28,29].

In Pakistan, marble is considered among the top extracted minerals [30]. Pakistan has also gifted with 160 million tons of marble deposits [22,30]. Marble and stone industry has grown rapidly in Pakistan with extraction of 3000 tons of marble in 1960s to approximately 450,000 tons in 1990s [31,32]. Growing industry also raised the amount of generated waste. This waste can pollute the environment [22]. WMP being light weight can be transported through air and causes the contamination of natural reservoirs [24,33]. There is no feasible utilization of marble waste in present situation. Moreover, prolong exposure to environment having marble contaminants can lead to severe health issues [34-36]. Environment having marble effluents was reported as the source of kidney problems [13]. Moreover, WMP is also damaging agricultural lands leading to effect the crop generation [37]. Selfcompacting concrete can be prepared by utilizing marble as a filler material [38]. Effectiveness of WMP in improving the durability of concrete was explored by various researchers [31,39]. However, scant research is available regarding the utilization of WMP to control ASR. Focus of this study was to evaluate the effectiveness of WMP in controlling ASR expansion. Utilization of WMP in controlling ASR expansion can overcome the problem along with effective utilization of this abundant waste.

Table 1Mixture proportions of cement and WMP.

Mixture	Cement (%)	WMP (%)
REF	100	-
WMP10	90	10
WMP20	80	20
WMP30	70	30
WMP40	60	40

#### 2. Experimental program

To evaluate the suitability of WMP in controlling ASR expansion, experimental study was conducted in two phases. In the first phase, chemical and physical properties of raw materials were evaluated. Moreover, mortar mixtures (Table 1) incorporating WMP in various proportions were also prepared to determine the effect of WMP on fresh, mechanical and thermal properties of mortar mixtures. In second phase, role of WMP in controlling ASR expansion was evaluated following ASTM C1260 (Standard test method for potential alkali reactivity of aggregates). Microscopic and chemical behavior of selected specimens in controlling expansion due to ASR was also studied through scanning electron microscopy (SEM) and energy disperse X-ray spectroscopy (EDS).

#### 2.1. Materials

To study the effectiveness of WMP in controlling ASR, WMP in the form of slurry was obtained from the local marble industry located in Pakistan. Slurry was first oven dried at 100 °C for 24 h. Then, dried slurry was ground in a ball mill until all the particles were finer than 45  $\mu$ m, to be similar to cement particles. The obtained WMP was then used during the study. Local coarse aggregate with dolomite-limestone rock type from Pakistan was used to develop the phenomena of ASR. Dolomite-limestone rock type was observed alkali-silica reactive in previous studies [40,41]. Aggregate was obtained in desired sizes by blasting and wet crushing techniques. During the study, locally available ordinary Portland cement and clean tap water were used.

#### 2.2. Mixture proportions

Cement was replaced with WMP in various proportions (10%, 20%, 30% and 40% by weight) to prepare the mortar mixtures for cubes and bars. Cement to aggregate ratio was kept 1–2.25 to prepare the specimens. Constant (ASTM C1260) and varying water-cement ratios were used to prepare mortar bar specimens. Firstly, ASTM C1260 was followed and water-cement ratio was kept constant i.e., 0.47. For control mixture without WMP, flowability of 110 mm was obtained keeping constant water-cement ratio. However, with increasing proportion of WMP, decrease in flowability was observed. Therefore, mortar mixtures with varying water-cement ratio were also prepared to achieve the constant flow of 110 mm.

# 3. Test methodologies

#### 3.1. Raw materials

Chemical properties of raw materials (cement and WMP) were analyzed through X-ray fluorescence (XRF) and X-ray diffraction (XRD). However, physical properties of cement and WMP were determined through fineness and specific gravity. Furthermore, autoclave expansion of cement was also determined.

Petrographic examination (ASTM C295-Standard guide for petrographic examination of aggregates for concrete) and chemical test (ASTM C289-Standard test method for potential alkali-silica reactivity of aggregates (chemical method)) were used to determine the chemical composition of aggregate particles. However, aggregate physical properties were determined through water absorption, specific gravity, impact resistance, crushing resistance, abrasion resistance, bulk density, voids content and sulfate soundness.

#### 3.2. Fresh properties of mortar

Different ASTM standards were followed to determine the fresh properties of mortar mixture incorporating WMP in different proportions along with reactive aggregate. For all the mixtures consistency and setting time were determined following ASTM C187 (Standard test method for normal consistency of hydraulic cement) and ASTM C191 (Standard test methods for time of setting of hydraulic cement by vicat needle), respectively. European standard EN 196-3 (Methods of testing cement – part 3: determination of setting times and soundness) was used to determine the property of soundness. ASTM C1437 (Standard test method for flow of hydraulic cement mortar) was followed to determine the workability of mortar mixtures. First, flow was measured keeping watercement ratio constant i.e., 0.47 (following ASTM C1260). Generally addition of waste materials increases the water demand [42]. Therefore, for better understanding, change in water-cement ratio was also studied by keeping flow constant (i.e., 110 mm corresponding to 0.47 water-cement ratio). Workability is not directly linked with the effectiveness of WMP in controlling ASR expansion. However, better workability can be helpful for proper compaction leading to proper investigation of the suitability of WMP in controlling ASR.

# 3.3. Compressive strength

Mortar cubes of 50 mm  $\times$  50 mm  $\times$  50 mm incorporating WMP in various proportions were prepared and cured. Compressive strength of cubes was determined at different ages (7, 14, 28 and 56 days) following ASTM C109 (Standard test method for compressive strength of hydraulic cement mortars).

#### 3.4. Thermal examination

To study the thermal behavior of mortar mixture incorporating WMP, differential thermal analysis (DTA) and thermogravimetric analysis (TGA) were performed. Control mortar specimens and specimens with 10% of WMP after 28 days of curing were heated from 0 to 1100 °C to study the change in mass and mineralogical composition.

#### 3.5. Mortar bars expansion specimens

First, aggregate was graded following ASTM C1260 (Standard test method for potential alkali reactivity of aggregates (mortarbar method)). Mixing of mortar was done in accordance with ASTM C305 (Standard practice for mechanical mixing of hydraulic cement pastes and mortars of plastic consistency). Following ASTM C1260, three mortar bars (25 mm  $\times$  25 mm  $\times$  285 mm) for each combination were prepared using two gang prism molds. Change in length was measured in accordance with ASTM C490 (Standard practice for use of apparatus for the determination of length change of hardened cement paste, mortar, and concrete). Specimens were kept in an air tight container with 1 N NaOH solution at 80 °C for 28 days. At different days (7, 14, 21 and 28), change in length was measured. Furthermore, after expansion test, scanning electron microscopy (SEM) and energy disperse X-ray spectroscopy (EDS) were performed on the specimens to study change in microstructure and chemical composition.

#### 4. Results and discussion

#### 4.1. Material characterization

Table 2 shows the results of X-ray fluorescence (XRF) analysis of raw materials (cement and WMP). Presence of all the chemical compounds was observed within the specified limits [43]. However, presence of alkalies was observed higher than the limit. Amount of alkalies in cement should not exceed 0.6% to avoid ASR. In this study, it was observed equal to 0.84%. Generally, manufacturing process of cement is considered responsible for higher amount of alkalies [44]. Alkalies are considered as the main factor to cause ASR. According to ASTM C1260, alkalies in cement should be higher than 0.6% to study the ASR potential. Therefore, cement used during the study was appropriate with alkalies higher than 0.6%.

Presence of calcium oxide (CaO) was observed higher (i.e., 51%) in WMP. Amount of silica (SiO<sub>2</sub>) in WMP was also observed noticeable. WMP showed the presence of CaO in excess with magnesium oxide (MgO) in minor amount, which indicates the calcite form of WMP [45]. XRD results also confirmed later the presence of WMP

#### Table 2

Chemical composition of cement and WMP.

Components	Cement	Limits for cement [43]	WMP
SiO <sub>2</sub> (%)	20.8	17-25	6.56
Al <sub>2</sub> O <sub>3</sub> (%)	5.47	3–8	0.84
$Fe_2O_3$ (%)	3.53	0.5-6.0	0.24
CaO (%)	61.8	60-67	51.02
MgO (%)	2.97	0.5-4.0	1.12
Free lime (%)	1.01	<2.0	-
SO <sub>3</sub> (%)	1.57	2.0-3.5	0.09
Na <sub>2</sub> O (%)	0.45	_	0.41
K <sub>2</sub> O (%)	0.60	_	0.11
Na <sub>2</sub> O <sub>e</sub> (%)	0.84	<0.6	0.48
LOI (%)	2.11	<3	38.92
IR (%)	0.59	<0.75	-
°C₃S (%)	51.66	42-67	-
°C <sub>2</sub> S (%)	20.67	8-31	-
°C <sub>3</sub> A (%)	8.52	5-14	-
°C <sub>4</sub> AF (%)	10.74	6-12	-
LSF	0.91	0.66-1.02	-
SR	2.31	2–2.5	-
<sup>1</sup> AR	1.55	1.5-2.5	-

\* Based on Bogue's equation.

<sup>\*\*</sup> Lime saturation factor (LSF = CaO/( $2.8 \times SiO_2 + 1.2 \times Al_2O_3 + 0.65 \times Fe_2O_3$ ).

<sup>+</sup> Silica ratio (SR = SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub>).

<sup>¶</sup> Alumina ratio (AR =  $Al_2O_3/Fe_2O_3$ ).

in calcite form. Other compounds like alumina, ferric oxide, sodium oxide and potassium oxide were also observed in small amount. Loss on ignition (LOI) was observed 2.11% and 38.92% in cement and WMP, respectively. LOI was observed higher in WMP as compared to cement, which may be attributed to the loss of carbon dioxide [45]. Similar results were observed in previous studies [23,46].

Figs. 1 and 2 shows the results of X-ray diffraction (XRD) analysis of cement and WMP. Results showed that the presence of tricalcium silicate and dicalcium silicate was higher in cement. Presence of other compounds like tricalcium aluminate, tetracalcium alumino ferrite and calcium oxide was also observed in small amount. However, WMP showed the presence of calcite in major amount. Presence of quartz and corundum in minor amount was also observed in WMP. Presence of quartz and corundum showed that WMP can be the part of hydration process for the formation of calcium silicate gels as observed by Omar et al. [47]. Results of XRD analysis were observed consistent with XRF results.

Table 3 shows the physical properties of raw materials (cement and WMP). All the physical properties fulfil the ASTM specified limits. For instance, specific gravity of cement was observed 3.17, which was fulfilling the specified limit (3.10–3.25). WMP showed less unit weight and specific gravity than cement. Therefore, replacement of cement with WMP can result into reduced weight. Fineness of WMP was observed higher (3512 cm<sup>2</sup>/g) as compared to cement (2961 cm<sup>2</sup>/g). Therefore, WMP can be used as filler material to provide cohesion and micro filling in concrete [48].



Fig. 1. XRD results for cement.



Fig. 2. XRD results for WMP.

According to ASTM C1260, to evaluate the potential of ASR, autoclave expansion of cement should be less than 0.2%. In this regard, cement used during the study was appropriate with expansion of 0.043%. Results of physical properties of WMP were observed consistent with previous studies [23,48].

Chemical test results of aggregate source are presented in Table 4. Aggregate source showed the presence of calcium oxide (CaO), magnesium oxide (MgO) and silica (SiO<sub>2</sub>) in noticeable amount. Presence of other compounds like alumina (Al<sub>2</sub>O<sub>3</sub>), iron oxide (Fe<sub>2</sub>O<sub>3</sub>), potassium oxide (K<sub>2</sub>O) and sodium oxide (Na<sub>2</sub>O) was also observed in minor amount. Chemical composition of aggregate source showed that the source belongs to dolomite-limestone rock type [41].

Table 5 shows the physical properties of aggregate source. All the physical properties were observed within the ASTM specified limits. For instance, crushing value of aggregate source was observed equal to 21, which was less than the specified limit (i.e.,<25) in literature [49]. Aggregate source also showed better impact and abrasion resistance. Aggregate source being dolomite-limestone rock type showed less water absorption i.e., equal to 0.89% [50]. Therefore, based on physical properties aggregate source can be used in construction activities.

Petrographic examination of aggregate source is presented in Table 6. Results showed that aggregate source comprised of 53% dolomite and 47% limestone. Dolomite mineral comprised of anhedral to subhedral crystals with pore spaces. Spaces may be attributed to the change of calcite with dolomite. Texture of dolomite rock was observed idioblastic to porphyritic. Other minerals like quartz and calcite were observed in considerable amount. However, clay, pyrite, hematite, and organic matters were also observed in minor amount. Dolomite rock showed darker color on fresh surfaces whereas lighter color was observed on weathered surfaces. Limestone comprised of calcite in excess, however quartz and clay minerals were also observed in traces. Presence of hematite and limonite minerals was also observed in small amount. Clay minerals were observed in thin bands along with calcite. Texture of limestone was observed dark grey on fresh to light grey on weathered surfaces. Dolomite-limestone rock type was reported alkali silica reactive by Ghafoori and Islam [40,41]. Therefore, aggregate source was appropriate to evaluate the effect of WMP in controlling ASR.

#### Table 3

Physical characteristics of raw materials.

Table 4

Chemical composition of aggregate source.

Components	Aggregate source
SiO <sub>2</sub>	12.8
Al <sub>2</sub> O <sub>3</sub>	0.41
Fe <sub>2</sub> O <sub>3</sub>	0.25
CaO	43.7
MgO	13.4
Na <sub>2</sub> O	0.07
K <sub>2</sub> O	0.17
LOI	31.4

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Physical characteristics of aggregate source.

Physical property	Aggregate source	Limit [ASTM C33, [43]]
Specific gravity	2.61	>2.5
(ASTM C127)		
Water absorption (%)	0.89	<1
(ASTM C127)		
Impact value	14.7	<25
(BS 812-112)		
Crushing value	21.1	<25
(BS 812-110)		
Abrasion test	20.3	<50
(ASTM C535)		
Bulk density (kg/m <sup>3</sup> )	1572	1200-1760
(ASTM C29)		
Voids content (%)	35.94	33-42
(ASTM C29)		
Weight loss due to sulfate soundness (%)	1.15	<12
(ASTM C88)		

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Petrographic results of aggregate source.

Minerals	Dolomite group (53%)	Limestone group (47%)
Dolomite (%)	61	-
Calcite (%)	16	84
Quartz (%)	18	9
Clay (%)	1.5	2
Hematite (%)	0.5	1.5
Pyrite (%)	1	1.5
Limonite (%)	-	0.5
Organic matters (%)	0.5	1

4.2. Fresh properties of mortar mixtures incorporating WMP

#### 4.2.1. Consistency and setting time

Table 7 shows the results of consistency and setting time of WMP blended mixtures. It was observed that consistency increased after WMP replacement. For instance, consistency of cement paste after replacing 40% cement with WMP increased from 25% to 30%. Increased consistency may be attributed to high surface area of WMP [46]. Increase in setting time was also observed after replacing cement with WMP. Initial setting time was observed 92 min for control mixture with WMP; however it

Property	Cement	Limit	WMP	Standard
Fineness (Passing No. 200)	97.7%	>90%	100%	ASTM C 184
Fineness (Blaine air permeability)	2961 cm <sup>2</sup> /g	>2250 cm <sup>2</sup> /g	3512 cm <sup>2</sup> /g	ASTM C 204
Specific gravity	3.17	3.10-3.25	2.69	ASTM C 188
Unit weight (kg/m <sup>3</sup> )	1481	830-1650	1125	ASTM C 29
Autoclave expansion	0.043%	<0.8%	-	ASTM C 151

5	g	4
-	-	

Mixture	Standard consistency (%)	Initial setting time (min)	Final setting time (min)	Soundness (mm)	<sup>*</sup> Flow (mm)	Water-cement ratio
REF	25	92	167	1.6	110	0.47
WMP10	25.5	116.5	183	1.5	108	0.48
WMP20	27	148	218.5	1.5	107	0.49
WMP30	29	163.5	241	1.3	105	0.51
WMP40	30	172.5	269	1.2	101	0.53
Limit	-	>45	<375	<10	-	-

 Table 7

 Fresh properties of mortar mixtures incorporating WMP.

\* For constant water-cement ratio of 0.47 (in accordance with ASTM C1260).

<sup>¶</sup> For constant flow of 110 mm.

increased to 173 min after replacing 40% cement with WMP. Similarly, final setting time increased from 167 min to 269 min after 40% cement replacement with WMP. WMP is neither a pozzolanic material nor a fully inert material. Increase in setting time may be attributed to the partial-participation of WMP in hydration process [23]. Hydration process slows down with increasing content of WMP leading to increased setting time. Previous researches by Vardhan et al. [23] and Singh et al. [37] also showed the similar behavior of replacing cement with WMP.

#### 4.2.2. Soundness

Table 7 shows the results of soundness of cement after replacing WMP in various dosages. It was observed that soundness of cement decreased after cement replacement with WMP. For instance, soundness reduced from 1.6 mm to 1.2 mm after replacing 40% cement with WMP. Singh et al. [37] observed increase in soundness after replacing cement with WMP. Higher amount of magnesia in WMP as compared to cement was considered the reason behind increase in expansion. However, in current study amount of magnesia was observed less in WMP as compared to cement. Therefore, reduction in soundness was observed after replacing cement with WMP due to decrease in magnesia content. According to EN 196-3 standard, acceptable limit of soundness is 10 mm. All the mixtures satisfied the permissible limit of soundness.

#### 4.2.3. Workability

Table 7 shows the workability of mortar mixtures incorporating WMP. It was observed that the flow decreased with increasing WMP replacement. For instance, flow was observed 110 mm and 101 mm for control mixture and mixture incorporating 40% WMP in replacement of cement, respectively. Specific surface area of WMP was observed higher than cement. Therefore, WMP filled the spaces between cement particles leading to increased particle contact. As a result, internal friction increased, which resulted into decrease in flow. Rana et al. [27] also observed similar flow behavior with WMP.

Effect of replacing cement with WMP on water-cement ratio for constant flow of 110 mm is also presented in Table 7. Results showed that water-cement ratio increased to achieve the flow of 110 mm after replacing cement with WMP. For example, water-cement ratio increased from 0.47 to 0.53, after replacing 40% cement with WMP. High amount of fines resulted into increased water demand [37]. According to Rana et al. [27], replacement of cement with WMP resulted into decreased flow due to higher water demand. Similar results were observed by other researchers [51]. Therefore, it can be concluded that higher water is required to replace cement with WMP and to achieve proper workability.

## 4.3. Compressive strength

Fig. 3 shows the compressive strength results of mortar cubes incorporating WMP in replacement of cement. Strength of speci-



Fig. 3. Effect of WMP replacement on compressive strength of mortar cubes.

mens increased with time, which may be attributed to hydration process. For instance, strength of control specimens was observed 37.1 MPa at 7 days, which increased to 53.1 MPa after 56 days. It was observed that strength increased after replacing 10% cement with WMP. For instance, strength of control specimens was observed 48.9 MPa at 28 days and 53.1 MPa at 56 days: whereas mortar cubes incorporating 10% WMP showed strength of 49.4 MPa at 28 days and 53.2 MPa at 56 days. Increase in strength may be attributed to the filler effect of WMP. WMP can fill the pores leading to improve the microstructure of concrete resulting into improved mechanical properties [46]. According to Omar et al. [47], silica present in WMP can react with calcium hydroxide (Ca(OH)<sub>2</sub>), leading to increase the compressive strength of concrete. In the present study, amount of silica in WMP was observed 6.56%, which can contribute in increasing strength after reaction with Ca(OH)<sub>2</sub>. In previous study, Şahan [51] also reported substitution of 5–10% WMP in concrete helpful to improve the mechanical properties. WMP can also improve the binding property of concrete matrix through reaction of calcite with C<sub>3</sub>A [46]. WMP is neither a pozzolanic material nor a fully inert material. It can react with C<sub>3</sub>A in cement. Calcium carboaluminate is produced when CaCO<sub>3</sub> (calcite) from WMP reacts with C<sub>3</sub>A [52]. As a result of this reaction, binding of concrete matrix improves leading to increase the compressive strength.

On the other hand, strength decreased with increasing WMP replacement. For example, strength of control specimens was observed 53.1 MPa at 56 days; whereas mortar cubes incorporating 30% of WMP showed strength of 42.6 MPa at the same age. Decrease in strength may be attributed to the lower amount of C<sub>3</sub>A and C<sub>2</sub>S (required for hydration process) after replacing more than 10% cement with WMP [37]. Vardhan et al., [23] also observed the increase in strength for 10% cement replacement with WMP due to filler effect. However decrease in strength was reported when more than 10% cement was replaced with WMP similar to

this study. According to Şahan [51], 5–10% replacement of cement with WMP not only improves the mechanical properties, but also reduces the carbon dioxide emission during cement production by 12%. Moreover, Uysal and Yilmaz [53], reported that 2.5 US\$/ m<sup>3</sup> can be saved after replacing 10% cement with WMP. Therefore, based on the results 10% WMP can be effectively used in replacement of cement leading to sustainable and economical construction.

#### 4.4. Thermal analysis

Fig. 4 presents the differential thermal analysis (DTA) and thermogravimetric analysis (TGA) results of control mortar specimen and mortar specimen with 10% WMP. DTA curve of control mortar specimen (Fig.4(a)) showed an endothermic peak at 72 °C, which may be attributed to the removal of absorbed water. Second endothermic peak was observed at 589 °C due to the presence of



Fig. 4. Thermal analysis results of (a) control specimen and (b) specimen incorporating 10% of WMP.

Ca(OH)<sub>2</sub>. Moropoulou et al. [54] also observed Ca(OH)<sub>2</sub> presence at the temperature of 500 °C. The third endothermic peak was observed at 816 °C as a result of calcium carbonate decomposition. In previous studies, a lot of researchers reported the occurrence of decarbonation reactions between 700 to 900 °C [54,55].

TGA curve (Fig. 4(a)) of control mortar specimen showed a mass loss of 29%. From beginning to 700 °C, mass loss was observed decreasing gradually. However, sudden drop in TGA curve was observed between 700 and 900 °C. Control mortar specimen without WMP showed a mass loss of 25% between 700 and 900 °C. Aliabdo et al. [24] and Moropoulou et al. [54] also reported the mass loss due to presence of Ca(OH)<sub>2</sub> and calcium carbonate decomposition.

DTA curve (Fig. 4(b)) of mortar specimen with 10% WMP in replacement of cement showed an endothermic peak at 66 °C and 110 °C, owing to removal of hydration water. An exothermic peak was observed at 338 °C due to calcium silicate hydrates [56]. However, presence of  $Ca(OH)_2$  was observed by endothermic peak at 599 °C. Another endothermic peak due to decarbonation reaction was observed at 821 °C. Similar findings were observed in previous study [54]. From beginning to 700 °C, mass loss of 5% was observed between 700 °C to 1100 °C. TGA curve showed total mass loss of 30% for specimen incorporating 10% WMP in replacement of cement as a result of carbonate combustion.

Comparison of DTA curves of control mortar specimen and specimen with 10% WMP showed the formation of exothermic peak after replacing 10% cement with WMP. Calcium silicate hydrate may be considered as the reason behind the exothermic peak formation [54]. As Omar et al. [47] also reported that silica present in WMP can react with calcium hydroxide (Ca(OH)<sub>2</sub>) to form calcium silicate hydrate. From beginning to 700 °C, TGA curves showed a decrease in mass loss from 8% to 5% after replacing 10% cement with WMP. This reduction in mass loss also confirmed the consumption of Ca(OH)<sub>2</sub> to form calcium silicate hydrate. Reduction in Ca(OH)<sub>2</sub> to form calcium silicate hydrate was also observed by Abbas et al. [57] and Kazmi et al. [58]. A lot of researchers reported the lower amount of Ca(OH)<sub>2</sub> helpful in controlling ASR expansion [59,60]. However, specimen with 10% WMP showed mass loss of 30% in comparison with the mass loss of 29%, observed for control mortar specimen. This may be attributed to the decarbonation reactions after 700 °C. Marble being a carbonate rock, the replacement of cement by WMP resulted into higher amount of carbonates [27]. Results of thermal analysis were observed in accordance with the results of compressive strength.

#### 4.5. Mortar bar expansion results

Fig. 5 shows the mortar bar expansion results of specimens incorporating WMP in various dosages. Expansion of control specimens was observed 0.142% and 0.221% after 14 and 28 days, respectively. As per ASTM C1260, aggregate source can be considered as alkali-silica reactive if expansion observes more than 0.1% and 0.2% after 14 and 28 days, respectively. On the basis of observed results, aggregate source can be considered potentially reactive. Fig. 5 shows the reduction in mortar bar expansion after replacing cement with WMP. For instance, 28% and 50% reduction in expansion was observed for mortar bars with 10% and 40% WMP in replacement of cement, respectively. Highest reduction in expansion was observed for mortar bars incorporating 40% WMP. In previous study by Abbas et al. [57], similar 50% reduction in expansion was observed after replacing 40% rice husk ash with cement. However, in another study by Beglarigale and Yazici [61], 68% reduction in mortar bar expansion was observed after replacing 40% ground-granulated blast furnace slag with cement.



Fig. 5. Expansion of mortar bars containing WMP.

After 28 days, all the mortar bar specimens incorporating WMP showed expansion less than 0.2%. Previous studies reported the formation of calcium silicate hydrate (after replacing pozzolanic materials with cement) effective in reducing ASR expansion [57,58,62]. Calcium silicate hydrate being negatively charged takes all the alkalies leading to control ASR [59]. In the present study, compressive strength results and thermal analysis showed the formation of calcium silicate hydrate for mortar bars incorporating 10% WMP in replacement of cement. So mortar bars with 10% WMP in replacement of cement showed reduction in expansion due to formation of calcium silicate hydrate. Furthermore, amount of alkalies was also reduced after replacing cement with WMP. For instance, amount of alkalies was observed 0.84% in cement and 0.48% in WMP. Therefore, reduction in amount of alkalies played a key role in reducing expansion after replacing cement with WMP. In previous study, Thomas [10] also reported that ASR expansion can be controlled by reducing the amount of alkalies. Afshinnia and Poursaee [63] also observed the reduction in ASR expansion after replacing cement with waste clay brick powder. It was observed that 10% waste clay brick powder showed 35% reduction in ASR expansion. In present study, specimens incorporating 10% WMP in replacement of cement showed 28% reduction in expansion.

Water demand of mortar mixtures increased with increasing replacement ratio of WMP to achieve the constant flow of 110 mm (Table 7). Therefore in order to examine the effect of increased water demand on the performance of ASR distress, mor-



Fig. 6. Expansion of mortar bars incorporating WMP (at varying w/c ratio).

tar bars with increased water cement ratio (corresponding to a constant flow of 110 mm) were also examined. Fig. 6 shows the mortar bar expansion results of specimens with varying watercement ratio (to achieve constant flow of 110 mm). Reduction in expansion was observed after replacing cement with WMP. For instance, specimens incorporating 10% WMP and 40% WMP in replacement of cement showed expansion of 0.164% and 0.111%, respectively. Mortar bars incorporating 40% WMP showed highest reduction in expansion. Mortar bars with varying water-cement ratio showed more expansion (corresponding to flow of 110 mm) than mortar bars with constant water-cement ratio (i.e., 0.47). For example, mortar bars incorporating 20% WMP showed an expansion of 0.123% for constant water-cement ratio and 0.154% for varying water cement ratio. However, all the mortar bars incorporating WMP in replacement of cement showed expansion less than the ASTM C1260 specified limit for reactiveness. Generally, shrinkage occurs as a result of high water demand [64]. Mortar bars with constant water-cement ratio showed decreased expansion as result of shrinkage due to high water demand. In previous studies, ASR expansion was also controlled using other waste materials [11,62,63,65]. Afshinnia and Rangaraju [66] also observed the formation of cracks along with longitudinal deformation in control mortar bar specimens due to ASR.

In this study, smaller surface cracks were observed in control mortar bar specimens. However, specimens showed no signs of deformation. No cracking and deformation was observed in mortar bar specimens incorporating WMP. Therefore, based on results WMP (up to 40%) in replacement of cement can be effectively used to control ASR distress.

#### 4.6. Microscopic examination and EDS analysis

Control mortar bars and mortar bars with 10% and 20% WMP replacement were examined through scanning electron microscopy (SEM) after immersion in 1 N NaOH solution for 28 days at 80 °C. Fig. 7 shows the SEM images of control mortar bars. Cracking was observed in control specimens, which may be attributed to ASR expansion (Fig. 7(a)). Initially, after casting and during examining, no cracking was observed. Cracks appeared after placing control specimens in 1 N NaOH solution for 28 days at 80 °C. Size of the cracks due to ASR expansion in control specimens was observed 10  $\mu$ m (Fig. 7(b)). In previous study by Kazmi et al. [58], control mortar bars also showed 10  $\mu$ m size cracks due to ASR expansion. However, no presence of ASR gel was observed in SEM images of control specimens. In previous studies, Beglarigale and Yazici [61] and Abbas et al. [57] also reported the formation of cracks without gel due to ASR expansion.

Microstructure of mortar bar specimens with 10% WMP in replacement of cement is presented in Fig. 8. No signs of cracking and ASR were observed after replacing 10% cement with WMP (Fig. 8(a)) owing to formation of calcium silicate hydrate and reduced alkali content. Mortar bar specimens incorporating 10% WMP showed dense structure (Fig. 8(b)), in accordance with the results of compressive strength test. Mortar bar specimens incorporating 20% WMP in replacement of cement also showed no signs of ASR and cracking (Fig. 9(a)) owing to reduced alkali content, in accordance with mortar bar test results. However, voids and pores were observed in specimens after replacing 20% cement with WMP (Fig. 9(b)). Ettringite needles were also found in the pores. Aliabdo et al. [24] also observed the ettringite needles in marble dust blended cement paste. In previous study, Abbas et al. [57] also observed no indication of ASR product in mortar bars after replacing 20% rice husk ash with cement. Moreover, Rana et al. [27] also observed dense microstructure of concrete after replacing 10% cement with WMP. On the basis of microscopic images, it can be concluded that WMP can be effectively used to control ASR



Fig. 7. (a) and (b) – Microscopic images of cracking in control mortar bars without WMP due to ASR.



specimen

specimen

Fig. 8. Microscopic images of WMP10 mortar bars.



(a) No signs of cracking observed in WMP20 specimen

(b) Porous structure observed in WMP20 specimen

Fig. 9. Microscopic images of WMP20 mortar bars.

Table 8

expansion. Moreover, dense structure was observed after replacing 10% cement with WMP, which improved the compressive strength.

Energy disperse X-ray spectroscopy (EDS) was performed to study the change in chemical composition of control specimens and specimens with 10% WMP replacement after immersion in 1 N NaOH solution at 80 °C for 28 days. Results of EDS analysis are presented in Table 8. Control specimens showed lower amount of SiO<sub>2</sub> in comparison with specimens incorporating 10% WMP in replacement of cement. For instance, control specimens showed amount of SiO<sub>2</sub> equal to 34.29%, which increased to 35.45% after 10% cement replacement with WMP. Similarly, amount of CaO was observed higher (i.e., 52.51%) after replacing 10% cement with WMP as compared to control specimens (i.e., 53.04%). However, CaO/SiO<sub>2</sub> showed no considerable effect of replacing 10% cement with WMP. Increased amount of SiO<sub>2</sub> may be attributed to the for-

Results of EDS analysis.	
Components	

Components	Mass present/ratio of mass present		
	С	WMP10	
SiO <sub>2</sub> (%)	34.29	35.45	
Al <sub>2</sub> O <sub>3</sub> (%)	5.17	4.81	
CaO (%)	52.51	53.04	
Na <sub>2</sub> O (%)	15.12	10.17	
K <sub>2</sub> O (%)	3.11	0.91	
Na <sub>2</sub> O <sub>e</sub> (%)	17.16	10.76	
CaO/SiO <sub>2</sub>	1.53	1.54	

mation of calcium silicate hydrate as observed in thermal analysis. Marble being calcite nature increased the amount of CaO in concrete. In previous studies, Vardhan et al. [23] and Kirgiz [67] also observed the increased amount of CaO after replacing cement with WMP.

Amount of alkalies reduced after replacing cement with WMP. For example, amount of alkalies were observed 17.16% for control specimens, which reduced to 10.76% after replacing 10% cement with WMP. Chemical composition of cement and WMP showed lower amount of alkalies in WMP (0.48%) as compared to cement (0.84%). Therefore, amount of alkalies reduced in concrete after replacing cement with WMP, leading to control ASR expansion. In previous study, Kazmi et al. [58] also observed the reduction in ASR expansion due to lower amount of alkalies after replacing sugarcane bagasse ash with cement. Based on the results, WMP can be effectively used to overcome ASR expansion.

# 5. Conclusions

Recycling WMP in concrete not only improves the durability of building materials but also reduces environmental burden. Based on the results, conclusions were drawn as follows.

- 1. WMP is neither a pozzolanic material nor a fully inert material. Reduced workability was observed for mixtures incorporating WMP. Mortar paste without WMP showed a flow of 110 mm; whereas, flow was 101 mm for mixture with 40% of WMP. Similarly, w/c ratio was increased from 0.47 to 0.53 after replacing 40% cement with WMP to achieve a constant flow of 110 mm.
- 2. Results of compressive strength showed increase in strength for specimens with 10% WMP at 28 and 56 days. However, higher content of WMP (i.e. 20%, 30% and 40%) showed a decrease in strength as compared to control specimens. Strength of control specimens was observed 48.9 MPa at 28 days and 53.1 MPa at 56 days; whereas mortar cubes incorporating 10% WMP showed strength of 49.4 MPa at 28 days and 53.2 MPa at 56 days. Therefore, based on the results 10% WMP can be effectively used in replacement of cement.
- 3. Mortar bar expansion reduced after replacing cement with WMP. Highest reduction in mortar bar expansion (i.e., 50% reduced expansion) was observed after replacing 40% cement with WMP, leading to overcome ASR distress.
- 4. Microscopic examination of control specimens showed the presence of cracks due to ASR. However, specimens incorporating WMP showed no signs of ASR cracking. Amount of alkalies also reduced after replacing cement with WMP, leading to reduce ASR expansion.

Therefore, WMP can be effectively used to control ASR expansion and to overcome related environmental issues leading to sustainable and economical construction.

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