

Article

# The Influence of Thermo-Mechanical Activation of Bentonite on the Mechanical and Durability Performance of Concrete

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**Abstract:** Despite presenting a very high global warming toll, Portland cement concrete is the most widely used construction material in the world. The eco-efficiency, economy, and the overall mechanical and durability performances of concrete can be improved by incorporating supplementary cementitious materials (SCMs) as partial substitutions to ordinary Portland cement (OPC). Naturally found bentonite possesses pozzolanic properties and has very low carbon footprint compared to OPC. By applying activation techniques, the reactivity of bentonite can be improved, and its incorporation levels can be maximized. In this study, the influence of mechanical and thermo-mechanical activation of bentonite is investigated on properties of concrete. Bentonite was used for 0%, 10%, 15%, 20%, 25%, 30%, and 35% mass replacements of OPC. Mechanical (compressive strength and split tensile strength) and durability (water absorption, sorptivity coefficient, and acid attack resistance) properties were studied. Results of experimental testing revealed that, concrete containing bentonite showed good mechanical performance, while durability was significantly improved relative to control mix. Application of thermo-mechanical activation can enhance the incorporation levels of bentonite in concrete. At 15% and 25%, bentonite produced optimum results for mechanical and thermo-mechanical activation, respectively. Bentonite inclusion is more beneficial to the durability than the mechanical strength of concrete.

**Keywords:** bentonite; compressive strength; sorptivity; acid attack resistance; durability of concrete; eco-efficient binder; low-cost binder

## 1. Introduction

In developing countries like Pakistan, the demands for housing and infrastructure development are increasing each day due to rapid urbanization and population growth. Being a major construction material, the demand for cement has increased abruptly in the recent past to fulfill the infrastructure

needs. The production of cement exceeded 4.1 billion tons in 2018 alone, which makes it the most used material after water [1]. Large scale, global, ordinary Portland cement (OPC) production has always been a big threat to the environment, resources, and economy. Nowadays, cement manufacturing accounts for more than 5% of worldwide CO<sub>2</sub> emissions [2]. Moreover, the cost of cement has increased almost 150% in the short span of 10 years [3].

The use of supplementary cementitious materials (SCMs) as replacements of OPC is of great interest for limiting the environmental impact of construction industry [4,5]. It is reported that CO<sub>2</sub> emissions can be minimized by replacing OPC with SCMs [6–9]. SCMs, which do not require any additional clinker processing, can significantly reduce the CO<sub>2</sub> emissions per ton of cementitious materials [4]. The use of natural pozzolans in binder or concrete can result in various beneficial properties, such as low hydration heat, improved later-strength, low permeability, high sulfate-attack resistance, and low alkali-silica reactivity [10,11]. It is estimated that concrete works in a building cost about 31% of the total, and OPC is the main cost-controlling constituent of concrete [12]. SCMs can be utilized for low-cost concrete construction as well [3,13].

Different types of SCMs such as fly ash, bentonite, metakaolin, silica fume, calcined shale, etc., have been used to improve the performance of cement-based composites [14–16]. Till now, SCMs have been extensively used (60%) in ready-mixed concrete owing to their pozzolanic nature [17]. SCMs give comparable results in terms of mechanical performance and show outstanding performances in terms of durability [10,18]. By blending SCMs with OPC, the penetration of harmful chemicals into concrete can be controlled. After the hydration of OPC, a primary by-product, calcium hydroxide (CH), is produced, which is the main target of corrosive media to deteriorate the concrete [19,20]. Supplementary pozzolanic material consumes the CH during the pozzolanic reaction and forms additional calcium silicate hydrate (C-S-H) gel, leading to a durable concrete [21–25]. Pozzolanic cement blends show resistance to thermal cracking due to its low heats of hydration. Moreover, these blended cements have improved ultimate compressive strengths and low permeability because of pore refinement [20,26,27].

In certain locations, natural SCMs or pozzolans are abundant and widely used in countries such as Italy, Germany, Greece, and China as supplements to OPC [28]. Natural SCMs have a wider range of composition and larger variability in physical characteristics compared to traditional or industrial SCMs. The applications of natural SCMs are limited and controlled by their local availability and competition with the industrially produced SCMs (by-products). In future, the use of natural SCMs will expand more because of their extensive deposits all over the world and proven technical advantages [29]. Clay minerals are hydrous aluminum phyllosilicates, composed of repeated two dimensional, tetrahedral (T) and octahedral (O), sheets in different ratios [30]. The two sheets are hold together by an exchangeable interlayer cation coordinated to H<sub>2</sub>O molecules in the interlayer region.

When T-sheets and O-sheets combine 2:1, the smectite group of clay minerals is formed. Bentonite clay (montmorillonite mineral) belongs to this group [31]. Bentonite is a natural pozzolana found in different regions of the world. The word bentonite was first proposed by Knight in 1898 [32]. Bentonite is also found in different areas of Pakistan, including the province Khyber Pakhtunkhwa [13]. Bentonite is basically an impure clay mainly consisting of montmorillonite mineral. Bentonite clay fulfills all the requirements of ASTM C-618 [33] to be used as a cement replacement material, as shown in Tables 1 and 2. Moreover, by heating this clay, its pozzolanic reactivity can be enhanced further compared to unheated condition [31,34]. By using bentonite clay as a supplement to OPC, an eco-friendly, economical, and durable concrete can be achieved [3,13].

Pozzolana can be activated by different methods; i.e., mechanical, thermal, and chemical activation. Mechanical activation means to ground the SCMs into powder form to increase their fineness and surface areas. Previous studies found that the pozzolanic reactivity of SCMs can be increased by mechanical activation (grinding) [35,36]. As in a pozzolanic reaction, the silica (SiO<sub>2</sub>) part of pozzolan reacts with CH in the presence of moisture and forms CSH gel. It was reported that the pozzolanic reaction is determined by rate of dissolution of SiO<sub>2</sub> in water, and the dissolution rate of SiO<sub>2</sub> is

proportional to its surface area [37,38]. Despite increasing surface area prolonging the grinding of a material, it also increases the number of active centers at the edges of the material structure which are more energetic than the normal structure; the pozzolanic reactivity of the material depends on the number of active centers [39,40]. Thermal activation means heat treatment (calcination). It can be heating of the pozzolan or the high temperature curing of the specimens made with pozzolans. It was reported that thermal treat of clays produce a metastable state due to structural disorder and an amorphous reactive structure is formed [41,42]. Different clays have different calcination effects on their pozzolanic reactivities. Mielenz et al. [43] studied thermal treatment effect on the pozzolanic reactivity of 70 different types of pozzolana and reported the optimum activation temperature range of 650–870 °C. In previous studies, the optimum activation temperature for Bentonite was reported as the range of 800–830 °C [28,31]. In our previous research [34], the optimum activation temperature of the bentonite clay was found to be 800 °C.

**Table 1.** Physical properties of ordinary Portland cement (OPC) and bentonite clay.

Physical Properties	OPC	Bentonite	ASTM C618 Class N Requirements (%)
% Retained #325 mesh	-	11.4	34 maximum
Blaine fineness ( $\text{cm}^2 \text{g}^{-1}$ )	3152	2571	-
Specific gravity ( $\text{g cm}^{-3}$ )	3.05	2.81	-
Average Particle size	20 $\mu\text{m}$	4 to 5 $\mu\text{m}$	-
Strength Activity Index (%)	-	-	-
7 days	-	84.4 [34]	75 minimum
28 days	-	85.3 [34]	75 minimum

**Table 2.** Chemical composition of OPC and bentonite clay.

Chemical Composition (wt.%)	OPC	Bentonite	ASTM C618 Class N Requirements (%)
Na <sub>2</sub> O	-	1.39	5 maximum
MgO	2.31	2.70	-
Al <sub>2</sub> O <sub>3</sub>	9.78	18.32	-
SiO <sub>2</sub>	18.8	56.6	-
K <sub>2</sub> O	-	0.67	-
CaO	59	3.1	-
TiO <sub>2</sub>	-	0.98	-
Fe <sub>2</sub> O <sub>3</sub>	3.44	6.1	-
SO <sub>3</sub>	2.85	-	-
(SiO <sub>2</sub> ) + (Al <sub>2</sub> O <sub>3</sub> ) + (Fe <sub>2</sub> O <sub>3</sub> )	-	80.93	70 minimum
Loss on ignition (LOI)	-	7.1	10 maximum

Many studies have been conducted in Pakistan for utilizing industrial wastes, such as bagasse ash [44], rice husk ash [45], fly ash [46,47], etc., as SCMs. Some natural pozzolana have also been studied [3,13]. In the existing literature, very few studies have been conducted to explore the potential of bentonite clay as partial replacement of cement. A study of bentonite on the mechanical and durability properties of concrete needs to be done in more detail. Memon et al., [13] studied sawabi bentonite clay (3%, 6%, 9%, 12%, 15%, 18%, and 21% by mass) as an OPC replacement and reported positive results of bentonite on the fresh and mechanical properties of concrete. Also, further study was recommended [13] to explore the mechanical and thermal activation of bentonite clay and to study its effects on the properties of concrete. Moreover, high replacement levels of bentonite clay need to be explored. This study explored the mechanical and thermo-mechanical activation of bentonite clay to enhance its incorporation level in concrete. The performance of concrete was studied, both for

mechanically activated bentonite (MAB) and thermo-mechanically activated bentonite (TMAB) (at the levels of 10%, 15%, 20%, 25%, 30%, and 35% by mass of OPC), in terms of compressive strength, split tensile strength, water absorption, sorptivity, and acid attack resistance up to 180 days. The results concluded that 15% mechanically-activated and 25% thermo-mechanically activated bentonite can be used (significantly) as substitutes of OPC, with good mechanical and even better durability properties for concrete.

## 2. Materials and Methods

### 2.1. Materials

#### 2.1.1. Binder

Ordinary Portland cement (OPC) Type-I was used in the research as a binder. Its physical properties and chemical composition are given in the Tables 1 and 2, respectively.

Bentonite clay taken from district Sawabi, KPK, Pakistan, was used in this research. The clay collected from the natural source was in crude form and could not be used directly in concrete, so after collection, the clay was transferred to ball mill machine to obtain the passing size of number 200 sieve with a nominal sieve opening of 0.074 mm and packed in polythene bags protected from light and moisture. Physical properties and the chemical composition of bentonite clay are illustrated in Tables 1 and 2, respectively. The Bentonite clay fulfils the requirements of ASTM C618-12 [33] to be used as natural pozzolana.

#### 2.1.2. Aggregates

Locally available Lawrencepur sand was used as a fine aggregate. Its properties are given in Table 3. Its particle size distribution as per ASTM C136-06 [48] is shown in Figure 1a. Margalla crush was used as coarse aggregate. Its properties are also given in Table 3. Its particle size distribution as per ASTM C136-06 is shown in Figure 1b.

Table 3. General properties of aggregates.

Property	Fine Aggregate	Coarse Aggregate
Maximum nominal size (mm)	4.75	22.5
Minimum nominal size (mm)	0.075	4.75
Saturated surface dry water absorption (%)	1.40	1.12
Fineness modulus	2.45	-
Abrasion value (%)	-	24.56

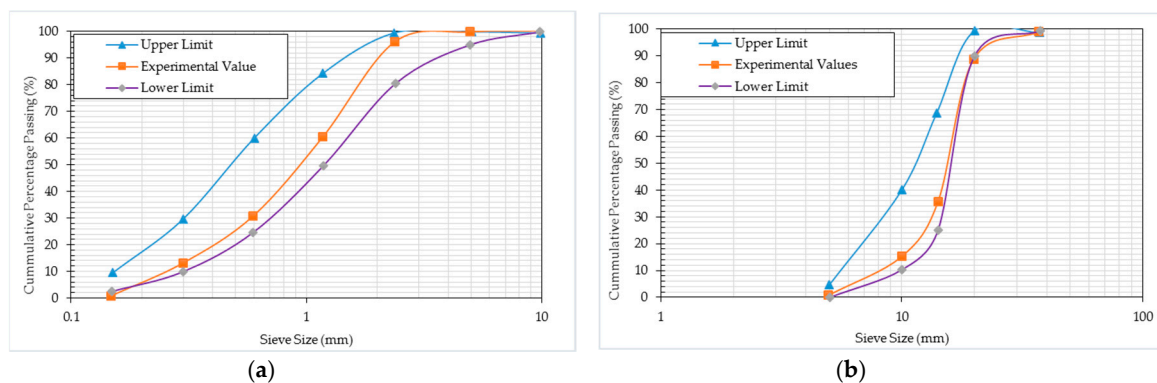


Figure 1. Particle size distribution of the (a) fine aggregate and (b) coarse aggregate.

## 2.2. Activation of Bentonite

### 2.2.1. Mechanical Activation (Grinding)

The clay collected from the natural source was first oven-dried for 24 h at 100 °C. After drying, the clay was ground in Loss Angeles abrasion machine. A total of 4500 revolutions were performed on each 5 kg batch of the clay to maintain uniform thickness; the powdered clay was then passed through sieve number 200 with a nominal sieve opening of 0.074 mm and packed in polythene bags to protect it from moisture.

### 2.2.2. Thermal Activation (Heating)

After mechanical grinding, bentonite clay was heated at 800 °C for 3 h. For control and uniform heating, the temperature was allowed to reach the target limit before placing the 5 kg clay batch in the furnace. Then the clay was put in the furnace for the required duration. The furnace took 24 h to reach room temperature. In the meantime, the sample remained in the furnace. The clay, after reaching room temperature, was packed into PVC bags in order to protect it from moisture.

## 2.3. Composition of Concrete Mixes

A total of 13 concrete mixes were produced in this study. Mechanically activated bentonite (MAB) and thermo-mechanically activated bentonite (TMAB) were used as 0, 10, 15, 20, 25, 30, and 35% by mass replacements of OPC. A constant water-cementitious material ratio of 0.5 was chosen for all mixes. The nomenclature and the composition of each mixture are presented in Table 4.

**Table 4.** Nomenclature and the compositions of mixtures.

Mix ID	Type of Activation	Bentonite by Weight of Cement (%)	Cement (kg/m <sup>3</sup> )	Bentonite (kg/m <sup>3</sup> )	Water (kg/m <sup>3</sup> )	Fine Aggregate (kg/m <sup>3</sup> )	Coarse Aggregate (kg/m <sup>3</sup> )
B0 (CON)	Control	0	425	0	213	635	1270
B10/G	MAB	10	382.5	42.5	213	635	1270
B15/G	MAB	15	361.25	63.75	213	635	1270
B20/G	MAB	20	340	85	213	635	1270
B25/G	MAB	25	318.75	106.25	213	635	1270
B30/G	MAB	30	297.5	127.5	213	635	1270
B35/G	MAB	35	276.25	148.75	213	635	1270
B10/G/T	TMAB	10	382.5	42.5	213	635	1270
B15/G/T	TMAB	15	361.25	63.75	213	635	1270
B20/G/T	TMAB	20	340	85	213	635	1270
B25/G/T	TMAB	25	318.75	106.25	213	635	1270
B30/G/T	TMAB	30	297.5	127.5	213	635	1270
B35/G/T	TMAB	35	276.25	148.75	213	635	1270

Where “B” denotes “bentonite”; “G” denotes “grinding”; “T” denotes “thermal”; “MAB” denotes “mechanically activated bentonite”; and “TMAB” denotes “thermo-mechanically activated bentonite”.

Mixing of all concrete mixes was done in a mechanical mixer of 0.15 m<sup>3</sup> capacity at the speed of 35 revolutions per minute. First, all solid ingredients were mixed for about 4 min. Subsequently, water was added, and mixing of concrete continued for the next 4 min. The total duration of mixing was kept to 8 min for all mixes.

## 2.4. Specimen Preparation and Testing

To assess the performance of concrete modified with bentonite clay, the following methodology was adopted. To evaluate mechanical performance compression and split tensile tests were performed. For compression testing, cylinders of 150 mm diameter and 300 mm height were cast as per ASTM C39 [49] for three different curing durations; i.e., 7, 28, and 180 days. For split tensile testing, cylinders of 150 mm diameter and 300 mm height were cast as per ASTM C496 [50] for curing durations of 7, 28,

and 180 days. For each test 3 specimens were cast and tested. The cylinders were demolded after 24 h of casting. After demolding, all the cylinders were cured in water for the required durations.

For durability assessment, water absorption, sorptivity coefficient, and  $H_2SO_4$ -acid attack resistance were determined. For sorptivity 50 mm thick and 100 mm diameter discs were prepared and tested as per ASTM C1585 [51]. Similarly, for water absorption, 50 mm thick and 100 mm diameter discs were tested as per ASTM C642 [52]. To evaluate acid attack resistance, for each mix a cubic specimen of 100 mm was cast and cured in normal water for 14 days to gain some strength. After 14 days each specimen was kept in 4% sulfuric acid ( $H_2SO_4$ ) solution for 7, 28, 56, and 90 days. To maintain 4% concentration, the acid solution was changed every week. The acid attack resistance was measured in terms of mass loss (%).

### 3. Results and Discussion

#### 3.1. Mechanical Properties

##### 3.1.1. Compressive Strength

Compressive strength is an important parameter of cement-based materials. Many durability and strength parameters of concrete can be predicted accurately using its compressive strength [53,54]. The compressive strength of each mix at 7, 28, and 180-days is presented in Figure 2. The general trend in results indicates that an increasing level of mechanically activated bentonite (MAB) reduces the compressive strength. The use of 10% MAB gives optimum results after 28 and 180-days compared to the control mix (CON). Compressive strength at all ages is badly affected by increasing levels of MAB. After 180 days, mixes with 10%–20% MAB gave better compressive strength than the strength of the control mix at day 28. The improvements in the compressive strength of concrete with the inclusion of 10% MAB can be ascribed to (1) its smaller particle size than OPC, which improves the particle size distribution in the binder matrix and (2) the reaction of bentonite particles with free CH (a primary hydration product) to produce secondary CSH-gels [21]. The reduction in compressive strength with an increasing incorporation level of MAB can be ascribed to reduction in overall CH content in the binder owing to lower calcium oxide percentage of bentonite which reduces the chance of MAB consumption in pozzolanic reaction. Naturally, bentonite is found in consolidated form; therefore, without a suitable treatment/activation method, it cannot be used directly in “as is” condition as a supplement for OPC. Memon et al., [13] reported that an MAB replacement of up to 21% does not affect the compressive strength of concrete compared to the control mix.

Concrete mixes with thermo-mechanically activated bentonite (TMAB) showed higher compressive strength than MAB mixes for a given age and replacement level. After 28-days, 10% incorporation of MAB and TMAB showed 1% and 4.3% higher compressive strengths than the control (CON), respectively, whereas, after 180-days, incorporation of 10% MAB and TMAB resulted in 3.5% and 9.8% higher compressive strengths than CON, respectively. Using the thermo-mechanical activation process, the replacement level of bentonite can be enhanced from 10% to 20% to produce a higher compressive strength than CON at 28-days. Clays may consist of some fixed crystalline structures and many of them possess pozzolanic properties. Calcination of a clay mineral alters its crystalline structure into an amorphous one and increases its pozzolanic reactivity considerably [55]. The heating of clay, shale or other mineral changes their structures to alumina or quasi-amorphous silica, which have good pozzolanic reactivities with CH [56].

Strength activity indices (SAIs) of all mixes at days 7 and 28 are displayed in Figure 3. ASTM C618 [33] states that for any material to be considered pozzolanic, the SAI should be at least 75% of the control blend at both day 7 and day 28. From the SAI values, it can be seen that MAB can be used up to 15% with a SAI higher than 75%. Whereas for TMAB up to a 25% level, SAI is well above 75% for both days 7 and 28. It can be concluded that thermo-mechanical activation produces mixes with more compressive strength than mechanical activation, which can help to maximize the level of bentonite in concrete production.



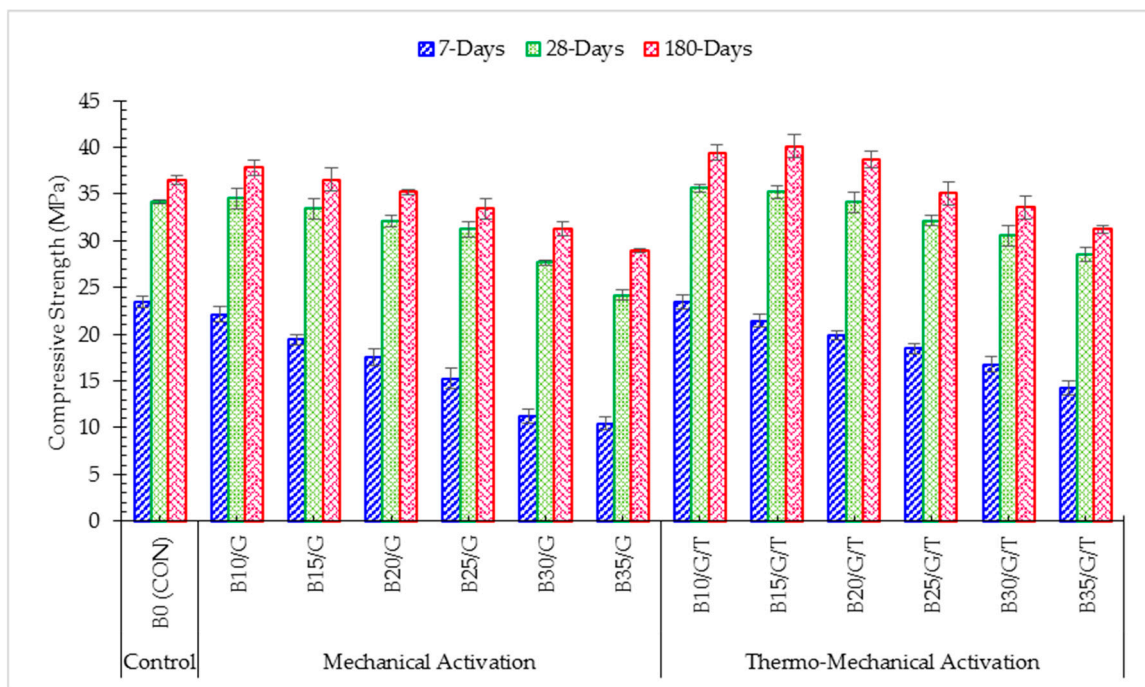


Figure 2. Compressive strength of each mix at 7, 28 and 180-days e clay.

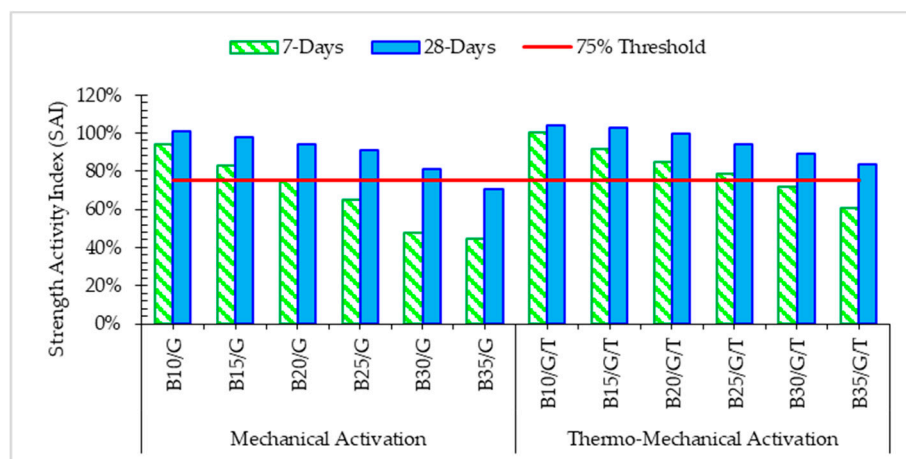


Figure 3. Strength activity indices of mixes after 7 and 28 days.

A relative analysis of compressive strength is also illustrated in the Figure 4. Compressive strength of the control mix (B0 (CON)) at day 28 was taken as the reference. It can be observed from Figure 4, that the day 28 compressive strength of concrete with 10%–20% MAB almost overlaps with the compressive strength of the reference mix and a notable decrease is noted for 20%–35% MAB. Similarly, for day 28's compressive strength of concrete modified with 10%–25% TMAB, it overlaps or nearly coincides with the compressive strength of the reference mix and a decline was observed for 25%–35% MAB. At day 180, mixes with 10%–20% MAB and 10%–25% MAB show significantly higher compressive strength than the reference.

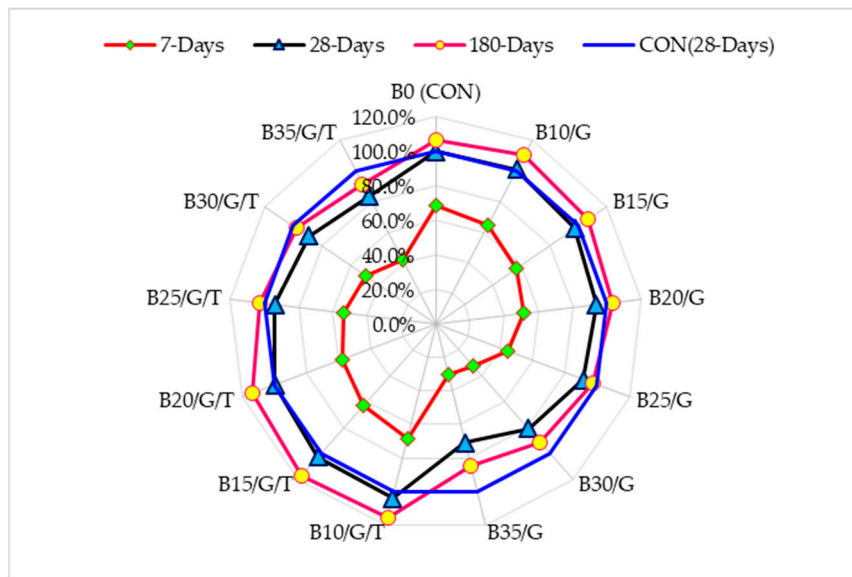


Figure 4. Compressive strength of each mix relative to the day 28 strength of reference mix (CON).

### 3.1.2. Split Tensile Strength

Results of split tensile testing are shown in Figure 5. At day 7, the tensile strengths of all MAB modified mixes were lower than that of the CON. Similarly to the compressive strength results, MAB incorporation of up to 10% produced maximal compressive strength after 28 and 180 days compared to CON. It is also worth mentioning here, that split tensile strength is more negatively influenced than compressive strength with increasing the incorporation of bentonite. This might be due to different natures of failure for the specimens under compression and split tensile tests. At higher levels of MAB, the filling effect of unreacted bentonite particles can play a good role in resisting compressive stresses, whereas, under splitting tension unreacted particles of bentonite may offer low resistance to failure (two failure surfaces can easily separate due to low cohesion in the binder); therefore, high levels of bentonite are more detrimental to tensile strength than compressive strength.

TMAB showed significant improvement over MAB, due to increased reactivity of clay minerals in pozzolanic reaction. Split tensile strength was reduced by 12% at a 35% level of TMAB after 28 days, whereas after 180 days, all TMAB incorporated mixes exhibited comparable or higher tensile strengths than the CON mix. MAB showed 3% and TMAB 5% higher split tensile strength than CON mix at day 180. But at higher levels, TMAB mixes performed 10% better than MAB. TMAB may have more reactive alumina and silica to react with CH, so have large pozzolanic reactivity [57,58]. Therefore, higher levels of TMAB show more strength development than those of the MAB.

Relative analysis of the split tensile strength results is presented in Figure 6. 28 days' strength of the control mix (B0 (CON)) was taken as the benchmark. At day 28, there is no notable surpassing from the loop of reference mix/benchmark. For mixes modified with MAB (10%–15%), the replacement overlapped the reference loop. For mixes modified with TMAB (at 10%–25% level), they coincided with the benchmark. At day 180, mixes with MAB (10%–15%) outperformed the reference mix, while all the mixes with TMAB, except B35/G/T, beat the reference mix and crossed the loop. Overall no significant difference was evident for the split tensile strength of TMAB incorporated concrete.



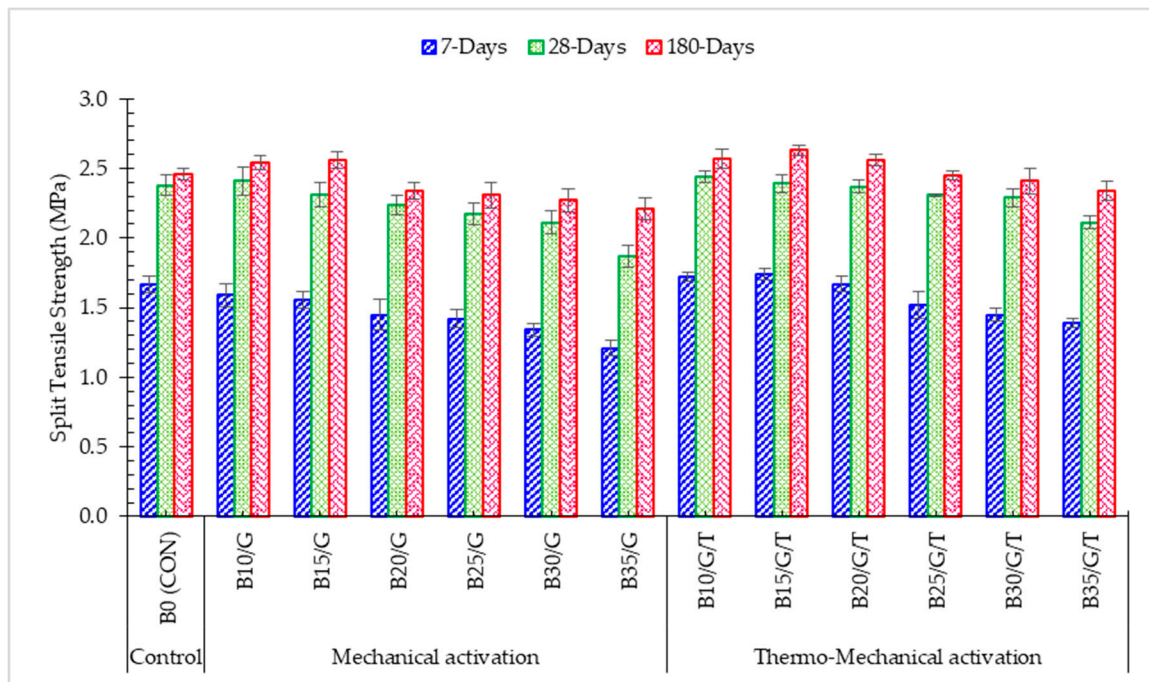


Figure 5. Split tensile strength of each mix after 7, 28, and 180 days.

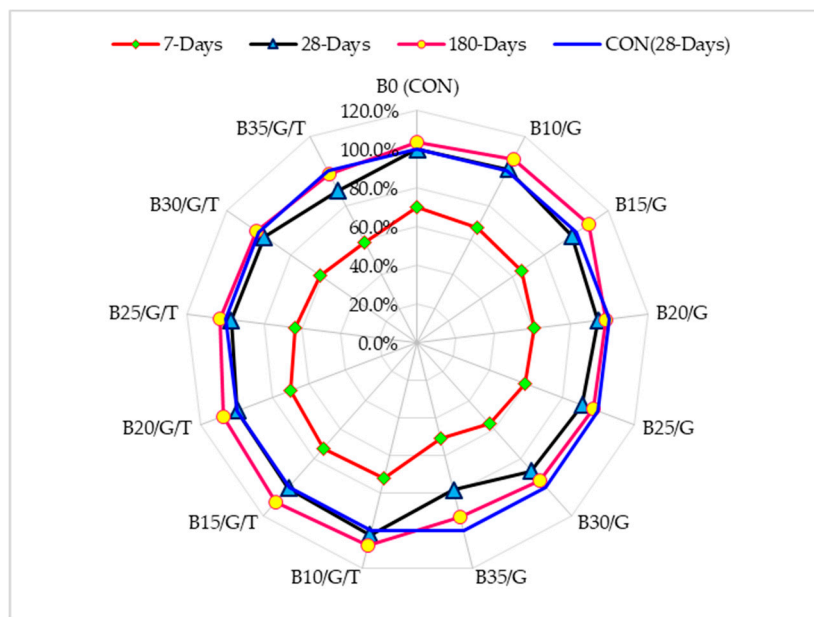


Figure 6. Split tensile strength of each mix relative to day 28's strength of reference mix (CON).

As, already mentioned, variation in compressive strength and split tensile strength with increasing incorporation level of bentonite follows a similar trend. Therefore, a strong relationship exists between compressive strength and split tensile strength. In Figure 7, the compressive strength of each mix at a given age and replacement level of bentonite is plotted against the corresponding value of split tensile strength. Equation (1) can be used to predict the cylindrical split tensile strength of concrete mix with a coefficient of determination ( $R^2$ ) higher than 0.9.

$$f_{ts} = 0.40 \times \sqrt{f_c} \quad (\text{for 7, 28, and 180 days, } R^2 = 0.92), \quad (1)$$

where  $f_{ts}$  (MPa) is split tensile strength and  $f_c$  (MPa) is compressive strength of cylindrical specimen.

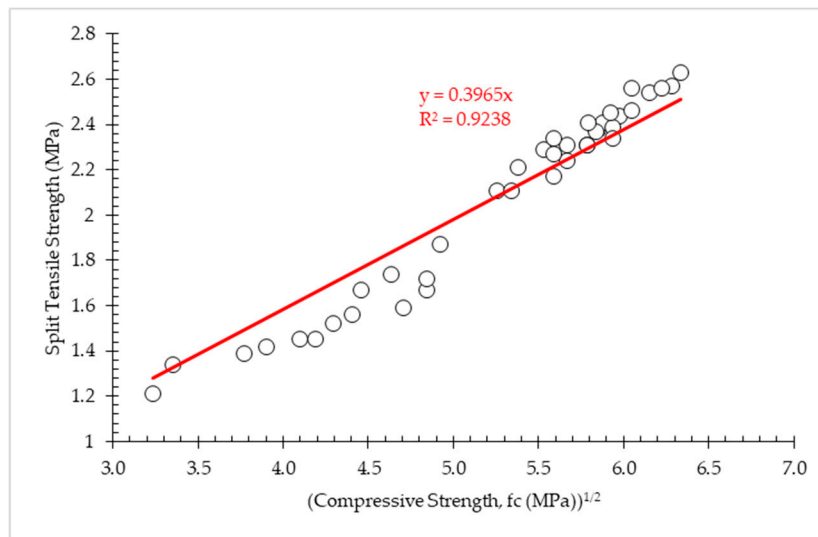


Figure 7. Correlation between experimental values of compressive strength and split tensile strength.

### 3.2. Durability Properties

#### 3.2.1. Water Absorption (WA)

WA is the measure of water accessible porosity of a material. WA is the indirect measurement durability of concrete, since water can carry harmful chemicals into concrete which may react with its constituents and result in changes in the properties of the material. WA tests were conducted on each mix after 28 and 180 days, and the results are given in Figure 8.

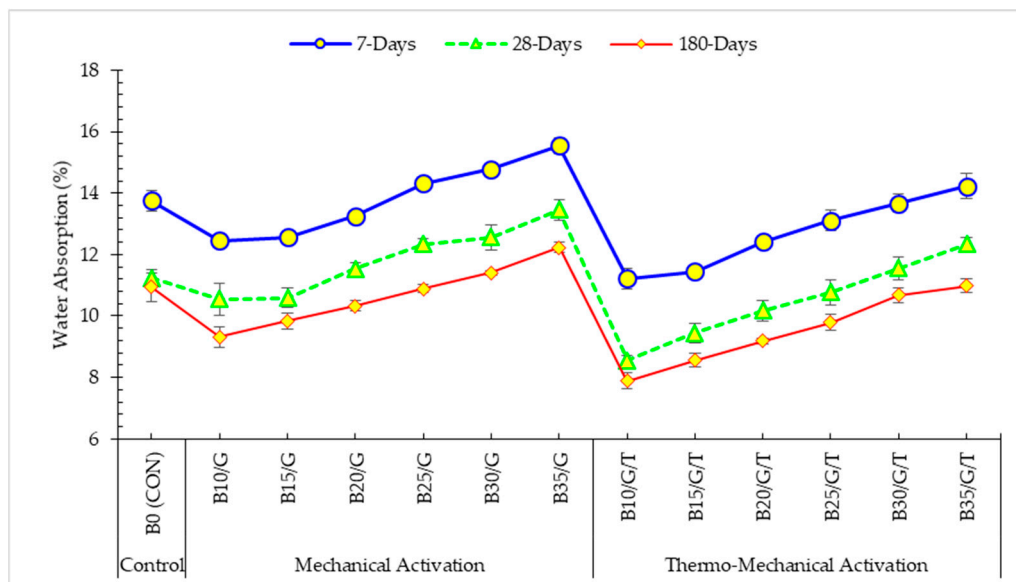


Figure 8. Water absorption test results after 7, 28, and 180 days.

A general trend indicates that the addition of 10%–15% MAB reduces the WA of concrete after 28 days, compared to CON. After 180 days, 10%–20% MAB reduces the WA of concrete compared to CON. This behavior can be ascribed to an improvement in particle size distribution which results in a reduction in effective pore volume of concrete. Reduction in pore volume results in reduction in WA at all ages, whereas chemical reaction between bentonite particles and free CH can decrease the length of interconnected pores. Studies [59,60] have reported that filler effect of pozzolans (fly ash, GGBS,

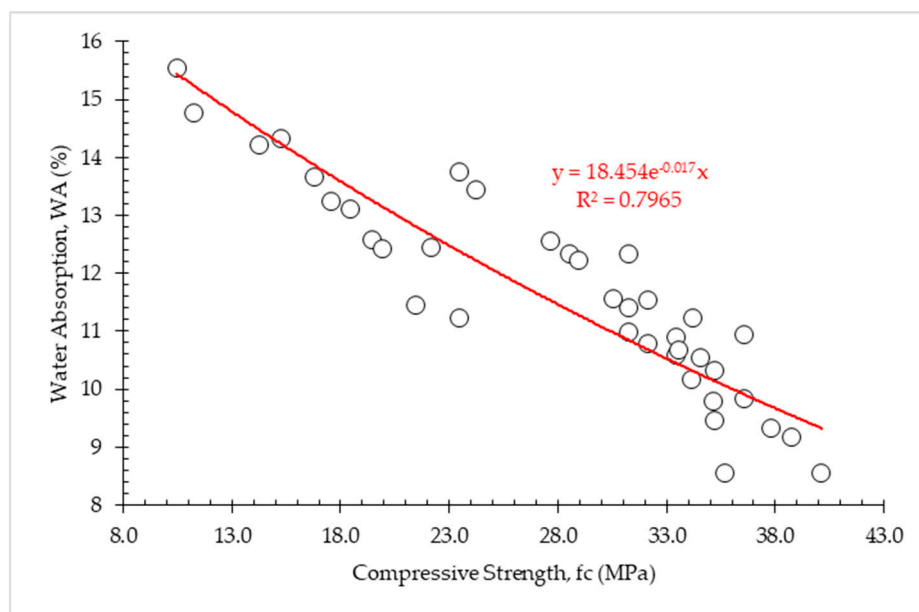
metakaolin, etc.) decreases the permeability of concrete, despite reducing the early age mechanical strength. After 180 days, the mix with 25% MAB shows WA comparable to that of the CON.

After 28 days, mixes with TMAB up to 25%, show higher resistance to WA compared to CON. After 180 days, a mix with any level of TMAB incorporated shows higher WA resistance than CON. This can be ascribed to improved reactivity of bentonite particles and better distribution of particles in the binder matrix. It was noticed that for a given age and replacement level of bentonite, TMAB-added mixes outperformed MAB by a notable margin. For example, at a 10% replacement of bentonite after 28 days, the MAB-added mix showed a 7% lower WA value compared to Con, whereas TMAB-added mix's value was 24% lower.

The experimental relationship between WA and compressive strength values is presented in Figure 9. Generally, compressive strength value increases with decreasing WA. An inverse-linear relationship is formed between two parameters, as given by Equation (2). The proposed relationship has good predictability, showing an  $R^2$  of 0.80.

$$WA (\%) = 18.45 \times e^{-0.017 \times f_c} \quad (\text{for 7, 28, and 180 days, } R^2 = 0.80), \quad (2)$$

where WA is water absorption in percentage, and  $f_c$  is cylindrical compressive strength of concrete in MPa.



**Figure 9.** Relationship between experimental values of compressive strength ( $f_c$ ) and water absorption.

### 3.2.2. Sorptivity Coefficient (SC)

SC can be defined as the absorption time rate of water by hydraulic cement concrete. It is one of the main properties of concrete for assessing its durability [61]. SC values are given in Figure 10. A similar trend like WA was observed for the results of SC with the varying incorporation levels of MAB and TMAB. For the same age and incorporation level of bentonite, TMAB mixes showed more resistance to sorptivity than MAB-added mixes. Since both WA and SC are functions of porosity and microstructural development of concrete, no significant difference in the results of SC and WA was observed with varying dosages of bentonite. After 180 days, almost all bentonite-added mixes showed higher resistance to sorptivity than CON, whereas after 28 days, up to 25% replacement of bentonite (either MAB or TMAB) with OPC lowered the sorptivity compared to the CON mix.

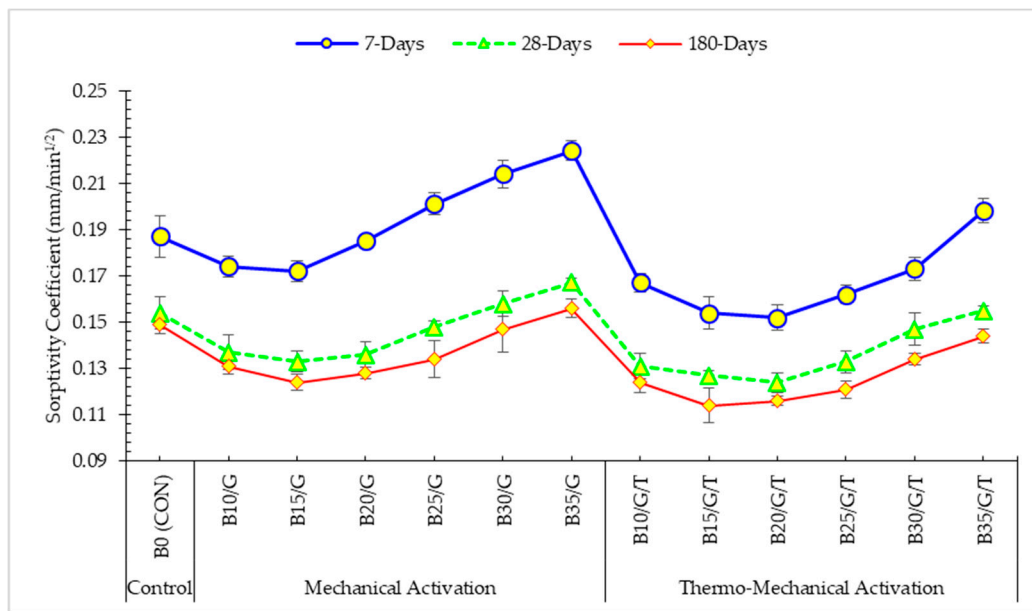


Figure 10. Sorptivity coefficient (SC) test results at days 7, 28, and 180.

Similarly to WA, SC has a good relationship with compressive strength; see Figure 11. Both SC and compressive strength are inversely related to each other in an exponential equation; see Equation (3). Since microstructural development and density due to bentonite inclusion leads to reduction in volume and connectivity of pores, these developments can also improve the compressive strength of concrete. Therefore, permeability-related durability and strength parameters are inversely related to each other.

$$SC \text{ (mm/min}^{0.5}\text{)} = 0.255 \times e^{-0.019 \times f_c} \text{ (for 7, 28, and 180 days, } R^2 = 0.85\text{)}, \quad (3)$$

where SC is sorptivity coefficient and  $f_c$  (MPa) is cylindrical compressive strength of concrete.

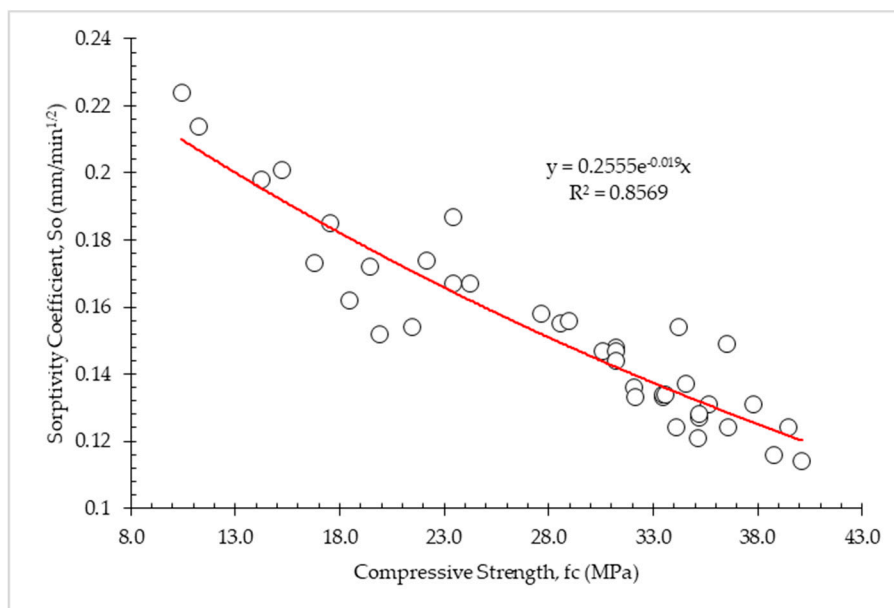
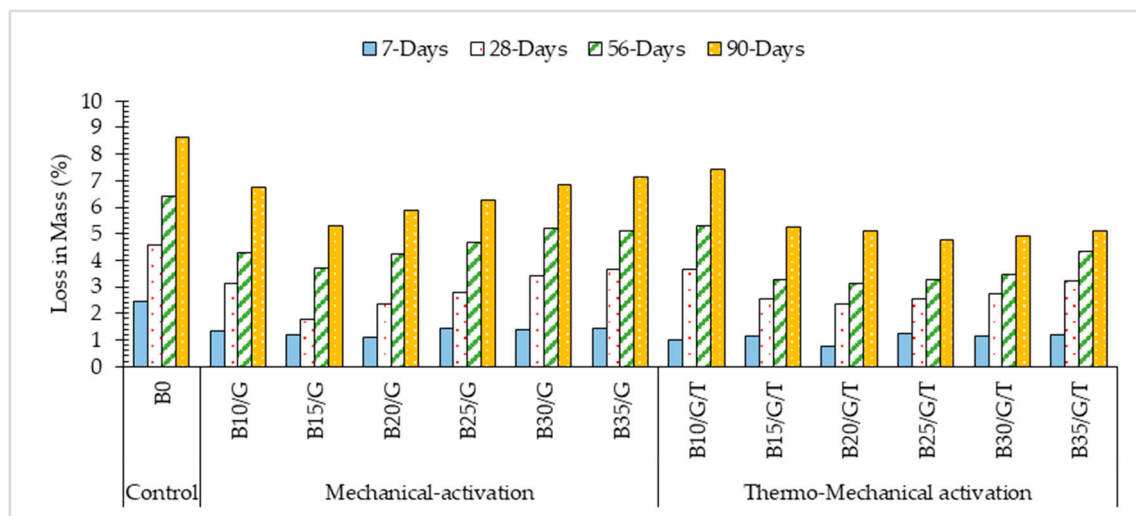


Figure 11. Relationship between experimental values of compressive strength ( $f_c$ ) sorptivity coefficient (SC).

### 3.2.3. Acid Attack Resistance ( $H_2SO_4$ Attack)

Acids have a strong tendency to react with the CHs of concrete in order to produce a calcium salt and water. There are many aggressive acids, such as acetic acids, nitric acids, hydrochloric acids, and sulfuric acids ( $H_2SO_4$ ). Concrete structures handling the acidic wastewaters of different industries are extremely vulnerable to deterioration. Therefore, it is very necessary to decrease the overall CH content of concrete. In this research,  $H_2SO_4$  was used for acid attacks on concrete mixes with various levels of MAB and TMAB.  $H_2SO_4$  reacts with CH and forms calcium sulfate which causes deterioration of the microstructure (due to increased internal pressure) and leads to loss in mass and mechanical strength of concrete. The results of acid attack are presented in terms of loss in mass of the specimens after 7, 28, 56, and 90 days for each mix, see Figure 12.



**Figure 12.** Loss in mass of bentonite-added mixes due to  $H_2SO_4$  attack.

The general trend in results indicates that the incorporation of either MAB or TMAB results in significant improvement in the  $H_2SO_4$  attack resistance of concrete. Increasing the incorporation level of bentonite not only consumes the CH in a pozzolanic reaction, but results in an overall reduction of the net CH content of binder [21,22]. OPC contains a very high content of CaO, whereas bentonite is rich in  $SiO_2$  and  $Al_2O_3$ , therefore, increasing MAB/TMAB level results in the reduction of CH (a primary hydration product of cement). It was also noticed that for a given age and incorporation level, TMAB-added mixes were more resistant to acid attack than MAB-added mixes. Not only does bentonite reduce the CH content of concrete, but it reduces the pore volume. Reduction in pore volume can also control the penetration of  $SO_4^{2-}$  ions from the solution into the microstructure that may cause the deterioration of concrete.

## 4. Conclusions

In this research, the influences on mechanical strength and durability of concrete, of various levels (0%, 10%, 15%, 20%, 25%, 30%, and 35% mass replacements of OPC) of mechanically activated (MAB) and thermo-mechanically activated (TMAB) bentonite clays, were investigated. The following conclusions can be drawn from the experimental results:

Compressive strength is badly affected when bentonite incorporation is increased beyond 25%. The maximum incorporation level of MAB is 10% to produce optimum performance after 28 and 180 days compared to the control mix and 20% MAB produces a comparable performance at 180 days. Contrastingly, maximum incorporation level of TMAB is 20%, in order to produce optimum performance after 28 and 180 days, and 25% to produce comparable results after 180 days. Split tensile strength



varies similarly to compressive strength with varying incorporation levels of bentonite. MAB/TMAB added mixes showed more strength development between 28 and 180 days than the control mix.

In general, more positive influence of bentonite incorporation was noticed on durability properties; i.e., WA, SC, and H<sub>2</sub>SO<sub>4</sub> attack resistance. Due to improved reactivity, TMAB mixes show notably improved durability properties over MAB mixes for a given level of bentonite. With an up to 20% level of bentonite, both MAB and TMAB mixes indicate high durability in terms of SC and WA after 28 and 180 days. All mixes incorporating MAB/TMAB, showed very high resistance to acid attack due to reductions in the overall CH contents of the concretes by both pozzolanic reactions and net reductions in CaO contents of binders.

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