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Properties of concrete mixes containing tire rubber and brick powder exposed to sulfuric acid and cured in water: A comparative study

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A R T I C L E I N F O

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Split tensile strength
Compressive strength

A B S T R A C T

The existing literature shows that rubberised concrete suffers from reduced mechanical properties when it is compared with normal density non-rubberised concrete. This is due to the underlying reduced bonding between tire rubber and other concrete ingredients. The massive sulfuric acid attack in rubberised concrete must have additionally discouraged researchers from attempts to assess the phenomenon of improving performance of rubberised concrete. A research was undertaken to compare the properties of concrete mixes containing tire rubber replacing coarse aggregate and waste clay brick powder (WCBP) replacing cement exposed to sulfuric acid and cured in water. Concrete cubes and cylinders of concrete grades of 20 MPa, 25 MPa and 30 MPa were immersed in 5% sulfuric acid solution up to 90 days following moist curing of 27 days. Other concrete cubes and cylinders were cured in water for comparison. The compressive strength findings indicated that all the specimens exposed to sulfuric acid had lost more than 57% of their compressive strengths after 90 days with reference to the corresponding samples cured in water. In contrast, out of all concrete mixes investigated for all concrete grades, never were the split tensile strength losses of the specimens exposed to sulfuric acid greater than 43.1% compared with those cured in water. In each exposure condition, concrete mixes with 5% WCBP showed slight improvements in compressive and split tensile strengths in contrast with the conventional concrete mixes. Visual inspection of the specimens illustrated depositions of flaky or white substances on the outer layers of specimens exposed to sulfuric acid compared with specimens cured in water. Moreover, the split tensile strengths of specimens were not severely affected with exposure to sulfuric acid in comparison with compressive strengths. Eventually, the research identified the existence of WCBP in rubberised concrete as a promising criterion of minimising strength losses of rubberised concrete.

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1. Introduction

The focus on the use of waste tire rubber (WTR) has allowed significant improvements of ductility properties of concrete and reduction of environmental pollution caused by dumping of waste tires [1–3]. However, challenges are being encountered in satisfactorily achieving improvements of other concrete properties due to incorporation of WTR within concrete. Considerable reductions in mechanical properties of concrete containing WTR have been observed in literature [4–13]. It is also known that the reductions in mechanical properties could be pronounced in rubberised concrete when exposed to adverse environments [14–16]. An example of these adverse conditions is sulfuric acid. On the other hand, researchers have found that incorporating waste clay brick powder (WCBP) in concrete enhances the mechanical performance of concrete [17–19]. Other researchers [20], have recommended on the introduction of a pozzolan to overcome reduced mechanical performance of rubberised concrete.

2. Literature review

Although concrete mixes are primarily based on good conditions, exposure to sulfuric acid could sometimes be inevitable in circumstances such as foundations of structures. Concrete structures including foundations are reported to be threatened by sulfuric acid attack from acid rain and ground water [21]. The oxidation of sulphide materials such as marcasites and pyrites is responsible for the formation of sulfuric acid in ground water [22]. In cases of sulfuric acid attack, the specimen surface degradation is initiated by the hardened binder matrix resolution [23]. The endurance of rubberised concrete immersed in sulfuric acid has suffered due to sulfuric acid attack [24]. To reduce the strength losses of rubberised concrete as a result of exposure to sulfuric acid, the utilisation of WCBP is proposed. The evaluation of properties of rubberised concrete with WCBP immersed in sulfuric acid is important. The replacement of ordinary Portland cement (OPC) with supplementary cementitious material (SCM) in concrete is advantageous for resistance from acid attack as this reduces the calcium amount of the hardened binder paste thereby decreasing the porosity of the disintegrated specimens [25]. Understanding of the variations happening within the hardened binder matrix in cases of acid invasion is vital towards concrete production having enhanced resistance from acid attack [25].

Industrial wastes are utilised as full or partial substitutes of coarse aggregate and fine aggregate. The production and crushing of the former is even faced with stricter restrictions in several countries [26]. Interestingly, the use of WTR aggregate of reduced graded particle sizes partially substituting fine aggregate has illustrated increased strengths and reduced water permeability in contrast with control concrete [27]. For this reason, it is known that coarse aggregates can be fully or partially substituted with vital materials. Various research works ([28–30], and the citations therein), have reported that recycled concrete aggregate, cockle shell, bamboo aggregate, waste glass and scrap tire chips could be utilised as replacements of natural coarse aggregates in concrete. Very interestingly, recycled aggregates replacing coarse aggregates have to some degrees shown satisfactory concrete performance in comparison with control concrete. Amongst the recycled aggregates, the utilisation of WTR aggregates has gained most recognition because of its satisfactory performance in ductility [17,19,31]. As the high demand of natural coarse aggregates has become apparent in construction sector [32], the substitution of coarse aggregates with WTR in concrete could be utilised to suppress the environmental pollution [12]. Moreover, the inclusion of WTR during concrete production is recommended for absorption of energy and vibration damping applications [33]. It is known that a substantial amount of WTR continues to be generated each day thereby posing environmental problems and disposal challenges [26].

The demand for OPC continues to escalate and since waste clay bricks are extensively produced in the construction industry, the associated pollution of waste clay bricks' dumping has generated renewed attention among researchers. Several studies are being undertaken in evaluating the opportunity of embracing the utilisation of brick powder in concrete as cement substitute material. It is known that concrete materials are extensively used in the construction industry. The vital concrete constituent, OPC, is largely responsible for the increased cost of concrete and results in carbon dioxide emission during its production [34]. The partial cement replacement with pozzolanic materials and reduction of production of cement originate from social and environmental challenges of energy conservation and sustainability. The partial replacement of OPC with materials of relevant pozzolanic characteristics is reported to be a breakthrough to challenges of depletion of natural materials and emissions of CO_2 into the atmosphere [35]. There are, of course, harmful consequences involved in dumping of waste and these not only contribute to spoiling of the atmosphere and land but also disturbing the outlook of urban surrounding [35]. Research has established that the utilisation of wastes for concrete production does not only decrease dumping challenges of waste materials but also reduces the cost of concrete [36]. On the other hand, pozzolans can be categorised into two types: natural and artificial pozzolans and these participate in a reaction with calcium hydroxide in the existence of water. There are strong indications that these aluminous and siliceous materials have no intrinsic cementitious constituents within themselves but when finely ground and in the presence of moisture, they undergo in a chemical reactivity with calcium hydroxide [37]. In spite of increased studies of WCBP and WTR and their recognition in modified concrete production extending back to the past decades, there remains a lack of a comparative research on performance of rubberised concrete with WCBP cured in water and submerged in sulfuric acid.

Sustainability in structural engineering remains the fundamental technique of reducing resources and energy consumption in construction and management of structures [38]. Partial replacement of OPC with alternative recovered materials can extensively decrease the environmental challenges in cement production, and hence, reduce production cost of concrete [17,39]. Research has illustrated that sulfuric acid attack on concrete could best be considered as sulphate attack under acidic environment [40]. It is reported that the combination of OPC, fly ash and silica fume usually shows optimum resistance to sulfuric acid invasion in concrete [41]. This approach has been observed to limit the lime and alumina contents of the cement matrix which is responsible for influencing the extents to which gypsum and ettringite might be created [40]. Studies covering pozzolanic materials have established some improvements of properties of concrete containing brick powder [42,43]. Chloride penetration and sulphate attack on concrete properties are observed to be reduced through inclusion of brick powder [42,44]. The reason perhaps lies in pozzolanic reactivity and micro-aggregate properties which are responsible for suppressing water permeability in concrete, optimising internal pore structure of concrete and increasing concrete compactness [45]. On the other hand, some existing studies have pointed out the shortcomings of rubberised concrete with respect to durability [7,15,16,46]. Sulfuric acid attack in rubberised concrete is established to be very detrimental in concrete performance [15]. This poor performance is attributed to not only the influence of dissolution from hydrogen ions but also sulphate ion attacks. The reduction in strength performance of rubberised concrete is owed to the weaker bonds within concrete constituent materials [46]. In order to establish improvement in rubberised concrete, introduction of pozzolanic materials could be considered [20].

Strength losses attributed to inclusion of tire rubber have been documented in literature [32,47–49]. Tire rubber has been found to negatively influence durability of concrete exposed to adverse environments [7,14,15,50]. Although literature is rich in exploring the performance of rubberised concrete submerged in sulfuric acid, few systematic studies have explored on performance of concrete mixes containing tire rubber and brick powder exposed to sulfuric acid. One approach to arrest the reductions in strength of rubberised concrete can be to use pozzolanic materials and such pozzolans can participate in pozzolanic reaction. Such pozzolanic reaction can probably arrest the compressive and mass losses of concrete from acid attack [51,52]. Other studies [53–57], have considered pre-treatment of tire rubber using prior treatment methods including the use of sodium hydroxide to improve its bonding properties. To shed more light on the performance of concrete mixes containing tire rubber and brick powder immersed in sulfuric acid in comparison with conventional concrete, experimental studies are essential. However, comparative studies on performance of concrete mixes containing tire rubber and brick powder exposed to sulfuric acid and cured in water have received less attention in literature. This study extends prior experimental works by the authors [58], on behaviour of rubberised concrete containing WCBP and is intended at evaluating behaviour of rubberised concrete containing WCBP exposed to sulfuric acid and cured in water.

3. Research significance

An important part of this research is the comparison of the performance of concrete mixes containing tire rubber and brick powder exposed to sulfuric acid and cured in water, still missing in literature, to develop a reliable methodology involving inclusion of brick powder in rubberised concrete. The aim of this investigation was to prepare brick powder from waste clay bricks and study its physical, chemical and morphological properties and possibly relate to pozzolanic reactivity. Since the pozzolanic reaction of materials should be the functions of strength gain in concrete and characteristics of pozzolans, the most part of pozzolanic reaction was studied by the gain in strength. In particular, such gain in strength could serve as a way of establishing reduced strength losses in rubberised concrete.

4. Materials and methods

4.1. Raw materials

The raw materials required for preparation of concrete mixes were OPC CEM I, brick powder, water, sand, coarse aggregates, tire rubber and sulfuric acid. WCBP was prepared by grinding fragmented clay bricks using a ball mill within the Department of Mechanical Engineering at Jomo Kenyatta University of Agriculture and Technology (JKUAT). The fragmented clay bricks which were ready to be dumped were collected at Kenya Clay Products. The pictorial summary giving insights on ball milling process is shown in Fig. 1. Coarse aggregate illustrated a specific gravity of 2.55 and was partly substituted with WTR. On the other hand, OPC was partly substituted with 5% WCBP with specific gravity of 2.69. OPC showing a specific gravity of 3.12, was of grade 42.5 in conformance with British cement specifications in the code [59]. In order to establish WCBP that can be utilised as a partial substitute of cement, WBCP passing

Fig. 1. The process of ball milling: (a) crushing fragmented clay bricks and (b) grinding crushed clay bricks using ball mill.

through 75 μm sieve was utilised. This WCBP demonstrated excellent pozzolanic properties specified in the code [37]. River sand was utilised in this study as fine aggregates according to the specifications in the standard [60]. The fine aggregate illustrating a specific gravity of 2.59 was procured from Meru region in Kenya. Tire rubber aggregates were produced from used tires collected from dumping locations of Nairobi and coarse aggregates were procured from Warren Concrete Company in Nairobi, Kenya. WTR aggregates and coarse aggregates were of sizes ranging from 5 mm to 20 mm in conformity with the specifications in the standard [60]. The gradations of coarse aggregate, fine aggregate and WTR are shown in Figs. 3–5 respectively. Non-conventional construction materials (WCBP and WTR) produced using waste materials are presented in Fig. 2. Portable water from JKUAT was used together with these constituent materials to make concrete samples.

4.2. Methods

4.2.1. Chemical compositions, mineral compositions and soundness experiments

Quantitative determination of soundness of cement was conducted using Le-chatelier method according to specifications in the code [61]. The process involves measurement of cement paste expansion following boiling. X-ray fluorescence (XRF) experiment evaluated the elements available in OPC and WCBP. These experimental works were carried out in Nairobi, Kenya at the Ministry of Petroleum and Mining. The experimental works employ current of 50–100 mA and voltage of 30–60 kV. X-ray diffraction (XRD) assessments of cement and WCBP were undertaken on a diffractometer (Bruker D2 Phaser) containing a graphite monochromator.

4.2.2. Scanning electron microscopy and image characterisation

The morphology and microstructure of OPC and WCBP were explored with a voltage system scanning electron microscope (SEM). The SEM characterisation process of scanning the specimens used the JEOL NeoScope JCM-7000 SEM model. Fig. 6 shows a JEOL NeoScope JCM-7000 SEM instrument including its structural components. Before performing the SEM experiments, the specimens were cleaned and dried in order to enhance exposure of surfaces. Specimens from OPC and WCBP were put on conductive adhesive tape and were positioned appropriately in the SEM machine. Afterwards, the images were generated when accelerated electron beams with low energy radiated the specimens and scanned the specimen surfaces. In an attempt to understand the structural parameters of WCBP and OPC, it was decided that image analysis-based methodology be employed. In this study, image analysis appeared to be a suitable technique in assessing the SEM images seeming to possess related properties. The particle areas, surface plots and surface roughness plots of OPC and WCBP were generated using Gwyddion and ImageJ software packages. The values of root mean square roughness (R_q) for WCBP and cement were computed using Gwyddion. Detailed processes for image analysis are reported in other studies [62,63].

4.2.3. Mix proportions

The mix ratios governing the compositions of OPC, sand and coarse aggregate in this research were developed to comply with requirements in the code [64]. The mix ratios were accomplished for 3 concrete grades of 20 MPa, 25 MPa and 30 MPa at day 28 in conformity with British research establishment (BRE). Procedures and mix design requirements are detailed in another study [65]. Mix ratios of 1:2.05:4.16 (OPC: fine aggregates: coarse aggregates) for 20 MPa concrete grade were generated using BRE technique of mix design. 25 MPa and 30 MPa concrete grades utilised mix ratios of 1:1.81:3.75 and 1:1.54:3.36 respectively. The conventional concrete mixes comprised OPC, sand and coarse aggregate all at 100%. Distinct from conventional concrete mixtures, the remaining concrete mixtures had 5% of WCBP and various quantities of WTR. For calculations in the latter case, coarse aggregates were partly substituted with WTR using volume substitution technique and OPC was partly substituted with WCBP by mass in conformance with other

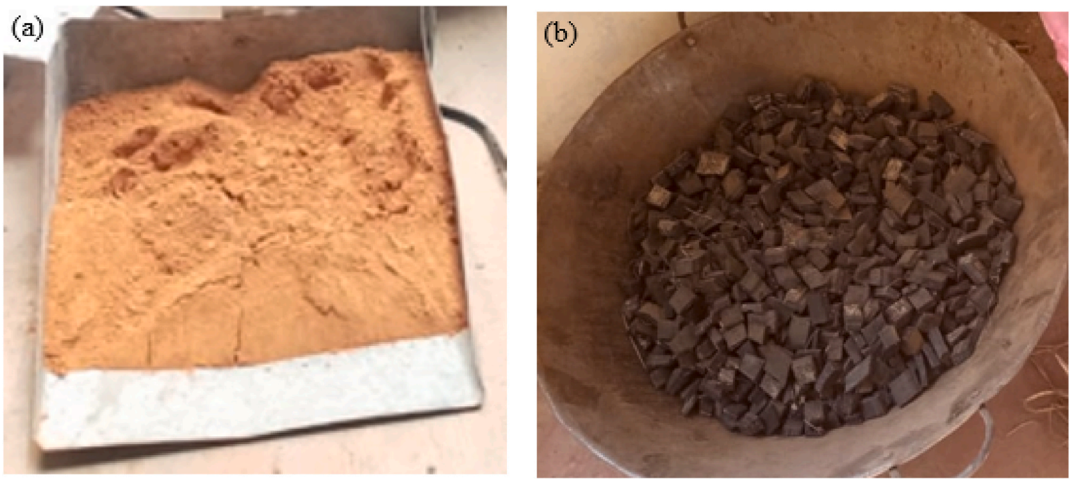


Fig. 2. Feasible non-conventional materials of construction (a) WCBP and (b) WTR.

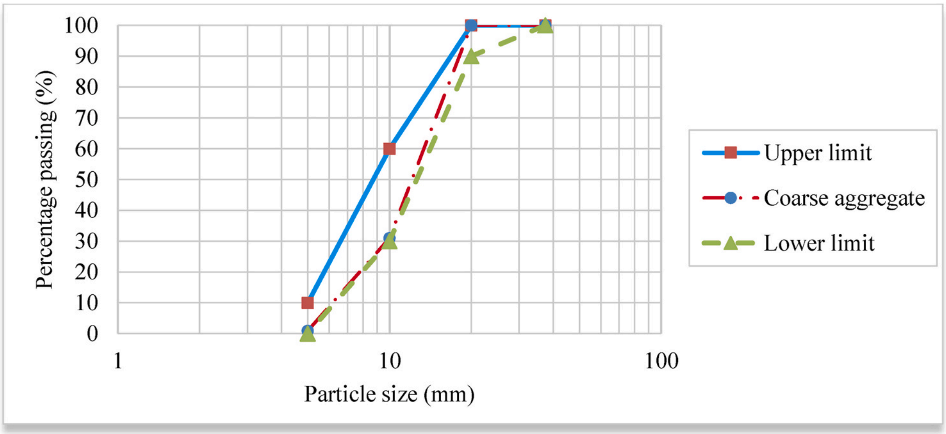


Fig. 3. Particle size distribution of coarse aggregate.

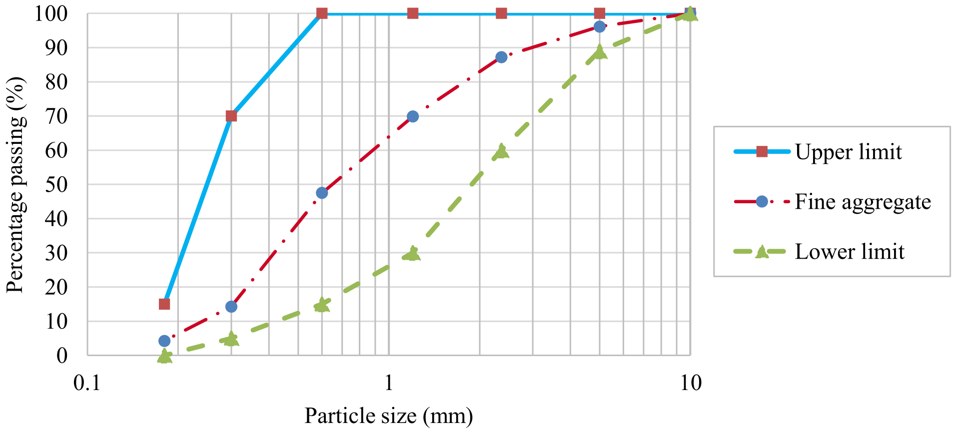


Fig. 4. Particle size distribution of fine aggregate.

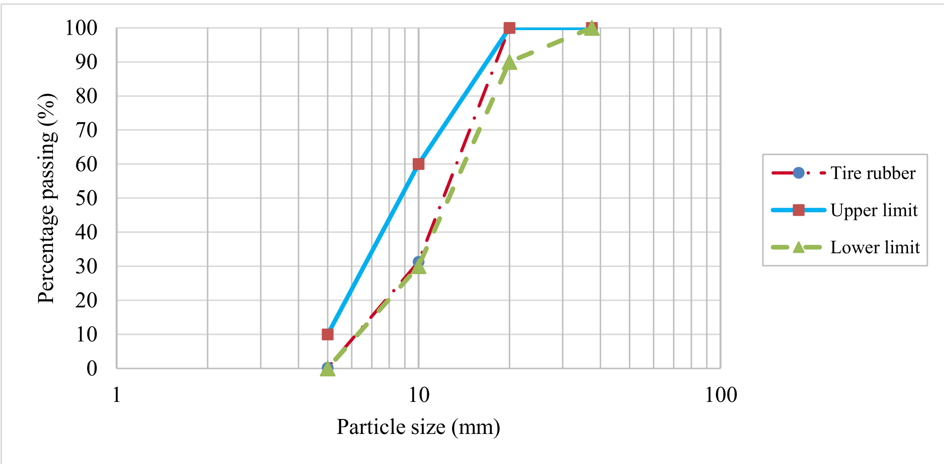


Fig. 5. Particle size distribution of WTR aggregate.

investigators [18,66]. The information about experimental matrix utilised in this research has been presented in Table 1. The following codes were used for the concrete mixes; 0P0T (conventional concrete), 5P0T (5% WCBP + 0% WTR), 5P10T (5% WCBP + 10% WTR) and 5P20T (5% WCBP + 20% WTR). In this study, the contents of 5% replacements of OPC with WCBP shown in Table 1 comprised

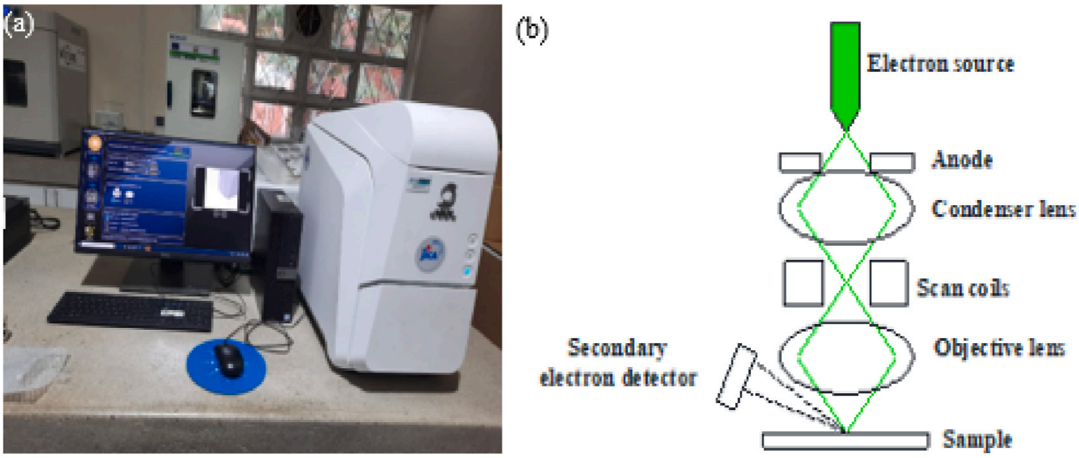


Fig. 6. (a) A picture of JEOL NeoScope JCM-7000 SEM equipment and (b) its structural components.

Table 1
Typical experimental matrix utilised in this research.

Class	Level			
	Control	Level 1	Level 2	Level 3
20 MPa	0% WTR +0% WCBP	0% WTR +5% WCBP	10% WTR +5% WCBP	20% WTR +5% WCBP
25 MPa	0% WTR +0% WCBP	0% WTR +5% WCBP	10% WTR +5% WCBP	20% WTR +5% WCBP
30 MPa	0% WTR +0% WCBP	0% WTR +5% WCBP	10% WTR +5% WCBP	20% WTR +5% WCBP

15.45 kg/m^3, 17.00 kg/m^3 and 18.89 kg/m^3 for 20 MPa, 25 MPa and 30 MPa concrete grades respectively. Formulated contents of 10% WTR substituting coarse aggregate utilised amounts of 50.87 kg/m^3, 50.42 kg/m^3 and 50.14 kg/m^3 for 20 MPa, 25 MPa and 30 MPa concrete grades respectively. Additionally, production of modified concrete with 20% WTR used WTR quantities of 101.74 kg/m^3, 100.85 kg/m^3 and 100.30 kg/m^3 for 20 MPa, 25 MPa and 30 MPa concrete grades respectively.

4.2.4. *Casting, mixing and curing concrete*

Mixing concrete was accomplished with trowels, metal plates, shovels and a concrete mixer with control mechanisms inhibiting losses of water. Using this method, a homogenous concrete was attained. To minimise concrete adherence to metal casting moulds, brushing was conducted prior to concrete placement in the moulds. Concrete mixtures were compacted using a poker vibrator with the goal of expelling air following casting of concrete in moulds. Trial attempts were conducted for all concrete grades to establish the recommended concrete mix ratios. A sum of 6 cylinders and 6 cubes were cast for every mixture. The cylinders of diameter of 100 mm and length of 200 mm and cubes of sizes of 100 × 100 × 100 mm were utilised during concrete placements in moulds. Cylinder and

Fig. 7. (a) Concrete curing in water and (b) concrete exposure to sulfuric acid.

cube specimens were taken out of the metal moulds after 24 h of concrete casting. Afterwards, the samples were placed in tanks with sulfuric acid and water (Fig. 7). The conditions were coded D1 (days in water) and D2 (days of moist curing and sulfuric acid exposure). The condition of D1 is the standard curing environment while D2 condition complied with specifications in the code [67]. Information on typical conditions of concrete curing and exposure is exhibited in Table 2. In the course of moist curing, concrete specimens were covered with wet gunny bags to make sure that the samples were adequately kept wet throughout the moist curing period. In this context, the significance of appropriate curing was emphasised. In the curing and exposure durations, temperature range for sulfuric acid and water was established as 21.7 °C–26.5 °C. It should also be noted that due to reduction of workability when WTR and WCBP are incorporated in concrete, additional water was necessary to concrete mixtures in order to maintain concrete workability. This is the key procedure to successful comparisons of results of concrete between control and modified concrete mixes [68].

4.2.5. Fresh concrete properties

Tests on fresh concrete properties were carried out with the use of slump test in compliance with the code [69]. The slump values used in this study conformed to the slump range of 10–30 mm in an effort to maintain concrete mixes' workability.

4.2.6. Measurements of compressive strengths

Concrete compressive strength experiments were undertaken in conformity with the code [70]. The experiments were carried out on 72 cube specimens following exposure to sulfuric acid and curing in water after 90 days. During the testing procedure, a universal testing machine (UTM) of range of 1500 kN was used. The cube specimens were aligned centrally on the UTM base plate prior to subjecting the specimens to loading. Loading was undertaken in such a way that buckling was avoided. Using a movable cross head, each specimen was subjected to the compressive load until collapse of the sample was recorded using load indicator. This compressive load was applied gradually at a rate of loading of 0.5 kN/s and the compressive strength value for each sample was generated from the maximum applied load divided by the cube specimen area in conformance with British standard of testing [71]. The mean compressive strength value obtained from three samples was presented.

4.2.7. Measurements of tensile splitting strengths

Tensile splitting strength tests were carried out on 72 cylinder samples according to the British standard of testing [72]. Just as with compressive strength tests, three specimens were evaluated for every mixture. Cleaning of the surfaces of the loading bearing rollers was undertaken to get rid of any materials deposited on the surfaces. Concrete cylinders which were cast and cured in water and exposed to sulfuric acid received diametrical loads from a UTM along the lengths of the cylinders. The applied loads at a rate of loading of 0.5 kN/s induced cracking on the specimens. To find the tensile splitting strength of every specimen, the maximum load which induced specimen failure was divided by parameters of specimen geometry as depicted in Equation (1).

$$\sigma_{ct} = \frac{2P}{\pi \times l \times d} \quad (1)$$

where d is the cross-sectional specimen diameter in mm, l is the specimen length (in mm) and P is the maximum loading (in kN).

4.2.8. Immersion in water and exposure to sulfuric acid

No ASTM standard is entirely dedicated to evaluate the concrete resistance to sulfuric acid invasion [24]. Therefore, the endurance of concrete specimens to sulfuric acid invasion was evaluated in conformance with the standard [67]. The standard is dedicated to resistance to chemical attack for polymer concrete, grouts and mortar [24]. 36 cylinders and 36 cubes for all mixtures were immersed in sulfuric acid of concentration of 5% soon after moist curing conducted for 27 days in nearly 98% relative humidity (Fig. 7b). Mixes with OPC and coarse aggregates substituted with WTR and WCBP respectively, were samples in addition to conventional concrete mixes. Prior to submerging in sulfuric acid, the specimen weights and diameters were measured. The duration of samples in moulds, moist curing and submerging in sulfuric acid was 90 days as shown in Table 2. The selected period is in compliance with specifications in the standard [67]. To ensure uniform distribution of the acid, the acid solution was stirred once in a week. The pH level was maintained at 2.4 ± 0.05 and its monitoring was conducted using a portable pH meter (Fig. 8b). Concurrent to preparation of specimens exposed to sulfuric acid, 36 cubes and 36 cylinders were prepared and cured in water for the duration of 90 days for comparison (Fig. 7a). The pH and temperature of water for curing the specimens were also measured (Fig. 8a). Subsequent to completion of 90-days duration, the specimens were removed from sulfuric acid and water. The samples exposed to sulfuric acid were gently washed in order to remove the loose constituents from the surfaces of the specimens. Afterwards, the specimens were subjected to 50% relative humidity condition for a period of 24 h. Diameter losses were measured using a ruler while the weight losses were determined using a weighing balance. The compressive strengths and split tensile strengths of the samples were also evaluated. In addition, visual inspection was conducted to compare the behaviour of concrete specimens cured in water and submerged in sulfuric acid.

Table 2
Curing and exposure conditions of concrete specimens.

Code	Days in mould	Days under moist curing	Days in water	Days in sulfuric acid
D1	1	0	89	0
D2	1	27	0	62

Fig. 8. Monitoring temperature and pH of exposure conditions using a portable pH meter for (a) samples cured in water and (b) samples exposed to sulfuric acid.

5. Results and discussion

5.1. *Chemical compositions, mineral compositions and soundness properties*

Table 3 enumerates the chemical elements available in OPC and WCBP. The table relates the percentage of every chemical element in OPC and WCBP. Figs. 9 and 10 indicate the XRD patterns of OPC and WCBP respectively. From Table 3, the dominating chemical is silica (SiO_2) and this is accompanied by iron oxide (Fe_2O_3). XRF studies indicate that the sum of the percentages of ($SiO_2 + Fe_2O_3 + Al_2O_3$) is 85.93% and is greater than 70% of the entire weight of WCBP suggesting full pozzolanic compliance with the code [37]. Literature suggests that if WCBP has substantial amount of SiO_2, then it has the opportunity to take part in pozzolanic reactivity when mixed with OPC. Sufficient time of residence of clay bricks in the furnace as well as burning of the bricks at temperature of 950 °C were in all probability the causes of substantial existence of silica in WCBP. The specification indicates that class N pozzolans show a maximum loss on ignition (LOI) and a minimum value of ($SiO_2 + Fe_2O_3 + Al_2O_3$) of 10% and 70%, respectively. Quantitatively, there appears to be substantial levels of SiO_2, Fe_2O_3 and Al_2O_3 in WCBP suggesting the feasible utilisation of WCBP as the OPC replacement in sustainable construction. High silica content in pozzolans suggests that the pozzolans could react with $Ca(OH)_2$ in cement to create calcium silicate hydrates (C–S–H) following hydration reaction. The LOI of cement slightly gives a different picture. According to literature [73], the LOI of cement should not exceed 5%. The high LOI might have occasioned from improper and prolonged storage or adulteration during transport or transfer. The XRF analyses also indicate that there were some additional unidentifiable chemicals in OPC and WCBP. This is evidenced by the sum of percentages of identifiable chemicals not being equal to 100% for both OPC and WCBP. What such unidentifiable elements could have been remains unknown and this study bears these out.

The desired soundness characteristics of OPC are reflected by the absence of MgO [74]. In this investigation, the soundness of cement was observed as 5.7 mm. Le-chatelier soundness values of not more than 10 mm also reflected in OPC in this research demonstrate that boiling the cement paste specimen does not provoke critical expansion [61]. It is reported that the use of OPC with enhanced expansion threatens the durability of structures [74]. The poor dimensional stability of cement after setting of OPC is observed to result in cracks which affect concrete structural member's durability [74]. There is poor dimensional stability in cases when excessive magnesium oxide content is present in cement and this content should be strictly controlled during cement production. The upper limit is reported as 5% in compliance with the code [59]. In this context, the lack of magnesium oxide in OPC could be an evidence that the OPC in this study was likely to exhibit desirable soundness properties. In addition to alumina and silica, which account for approximately 77.22%, this WCBP also contains small percentages of iron oxide, rutile, lime e. t.c. The iron oxide and alumina in WCBP illustrate vastly increased margins with silica but their combined proportions are observed to take part in a pozzolanic activity. Furthermore, the presence of rutile is demonstrated by reduced peaks and the significance of these characteristics has been mentioned by other investigators [75]. The occurrence of rutile is unsurprising as the compound is incorporated as a colorant when producing clay bricks in order to improve the mechanical behaviour of bricks [76].

Table 3
Chemical compositions of WCBP and OPC.

Material	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	MnO	P_2O_5	Ba	S	LOI
Cement	15.45	4.55	2.81	62.45	–	0.48	1.01	0.47	0.12	1.29	0.05	2.75	7.47
WCBP	64.36	12.86	8.71	2.00	–	1.82	3.05	2.13	0.68	1.18	1.18	–	0.97

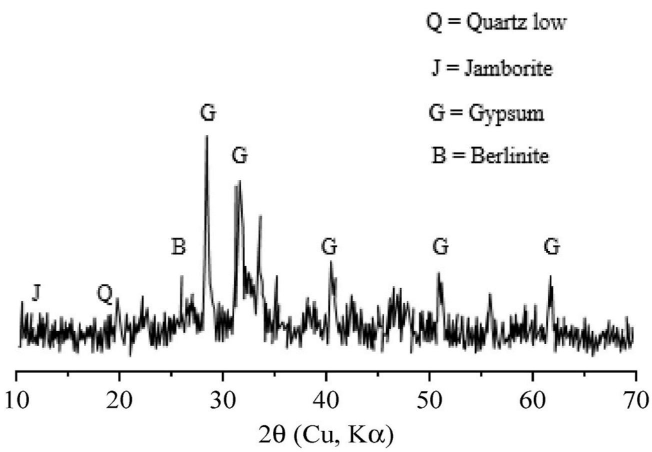


Fig. 9. The mineral constituents of OPC from XRD spectrum.

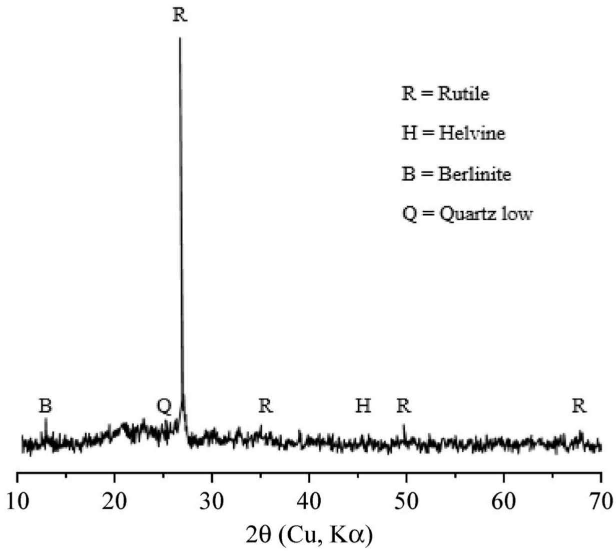


Fig. 10. The mineral constituents of WCBP from XRD spectrum.

The intensities in XRD are observed to exhibit the estimated compound amounts present in the specimen [77]. The existence of gypsum in OPC and silicates in WCBP is related with expected formation of calcium silicate hydrates (C–S–H) which is the cause of strength improvement. It is worth noting that the latter compound is strongly associated with pozzolanic reaction when combined with the former compound detected in the spectrum for OPC [78,79]. The creations of calcium monocarbonalumate taking place during the reactions of C_3A and carbonate ions [80,81], are examples which might be used to explain the properties of WCBP. In addition, since rutile is added during the production of clay bricks, the significant presence of this compound could not be surprising. Other investigators [82], have indicated that the presence of rutile in WCBP enhances mechanical behaviour of concrete. Surprisingly, the XRD analyses reflect that rutile in Fig. 6 is seemingly dominating. How this amount dominated in XRD remains unknown in this study and could be probably attributed to errors in XRD analyses compared with XRF analyses. From the XRF and XRD analyses, it seemed that it was difficult to get consistent data from both XRF and XRD. It could also be speculated that XRF findings are true representations of WCBP characteristics unlike XRD findings.

5.2. Scanning electron microscopy

The findings from SEM are shown in Fig. 11. Conventional SEM has great potential in direct characterisation of shapes of the particles and particle surface textures. The method is well capable of evidencing the particle size distributions, formation, morphology and sizes of materials by scanning samples on fine scaling [83,84]. Because of ease of operations, the method is preferred for the routine characterisation of the specimens, particularly solids [85]. In SEM, the electron beams having reduced energy are radiated into the specimens and are capable of scanning the surfaces of the specimens. The SEM investigation involved careful preparation of the

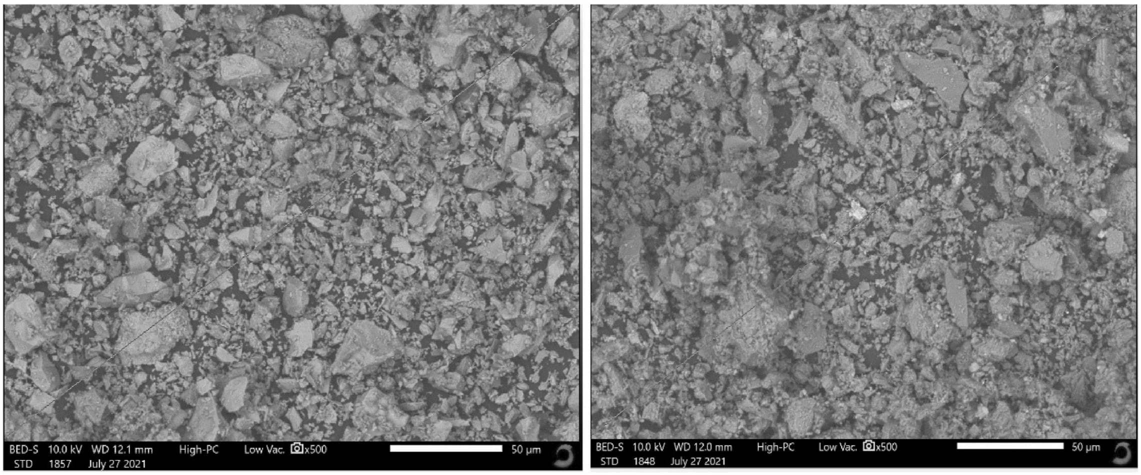


Fig. 11. Scanning electron microscopy of OPC (left) and WCBP (right).

samples intended at minimising errors during production of the images.

In Fig. 11, it is observed that the particles of WCBP in this research are neither spherical nor smooth. Nonetheless, the particles of WCBP illustrate irregular and sharp corners that are noticed to create slits. SEM image of WCBP seems to illustrate the occurrence of fine particles. A lot of fine particles are noticed to occur in slits and notches of large particles and these special arrangements have been noticed to enhance the water demand and decrease the workability of concrete incorporated with WCBP [76]. Although SEM micrographs are illustrated in Fig. 11, the similarity between SEM images of OPC and WCBP makes it difficult to evaluate the distinctions between the two micrographs. Currently, difficulties are met in adequately interpreting the morphological properties using SEM alone. The utilisation of image analysis presented in the subsequent sections seems to present more details about the morphological distinctions existing between cement and WCBP.

5.3. Image analysis

Image analysis could be used with greater confidence to obtain significant quantitative data from micrograph and transforms the micrograph into comprehensible information [86]. Image analysis can be applied once images are generated by microscopic tools such as SEM, optical microscopy (OP) and transmission electron microscopy (TEM) [86]. In this study, since differences between SEM images of cement and WCBP were difficult to discern, it was therefore decided to use image analysis in an attempt to identify quantifiable properties between the two samples.

5.3.1. Particle size and surface roughness parameters

Fig. 12 depicts the profiles of roughness of OPC and WCBP generated within diagonal lines shown in SEM micrographs in Fig. 11. Although the two roughness profiles appear to be similar, significant distinctions could be secured in the generated data for the profile

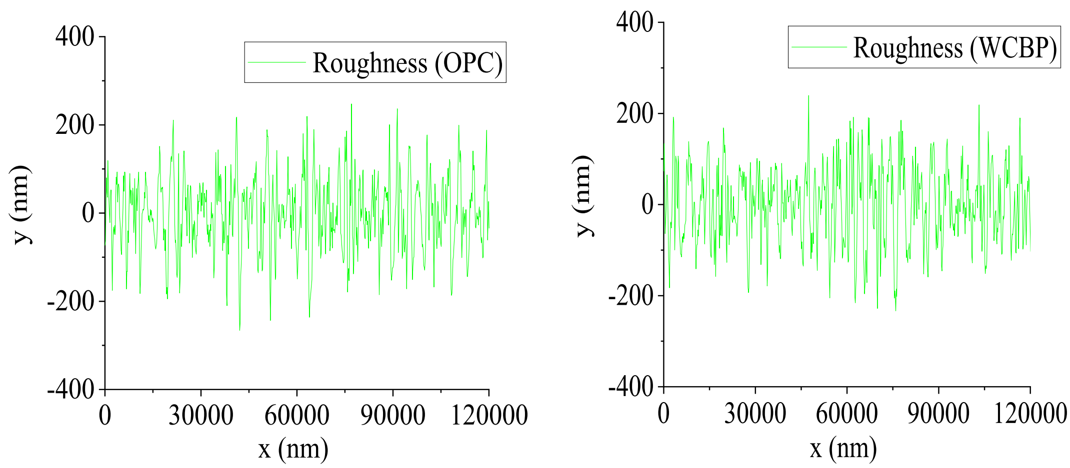


Fig. 12. Roughness curves of cement and WCBP generated using Gwyddion.

plots presented in Table 4. It should be mentioned that one of the methodological limitations is that the roughness parameters were generated along the diagonal line presented in Fig. 11. This decreased the probability of observing the roughness properties for the whole sections. Despite this limitation, it can be suggested that the generated roughness parameters could probably be representatives of other sections.

Despite similarities of SEM micrographs of cement and WCBP presented in Fig. 11, image analysis proved to differentiate the morphological properties between the two specimens. By applying ImageJ software, mean particle areas for OPC and WCBP were observed to be 1.27 μm^2 and 1.08 μm^2 respectively. From these findings, the fineness of WCBP can be illustrated and it is responsible at influencing the workability of concrete. Another possible interpretation of this observation is that during inclusion of WCBP in concrete, the pozzolanic reaction could be pronounced. The particle areas generated using ImageJ can be used to illustrate the fineness of WCBP, however, the SEM micrographs in Fig. 11 exhibit a better picture of their particle size distributions. The findings of reduced particle sizes combined with clear observation of reduced particles existing in notches and slits of larger particles previously explained, support the contention of increased water demand for concrete with WCBP. Other investigators [76], have established that the increasing fineness of WCBP imparted by enhanced grinding of fragmented clay bricks tends to impede the fresh concrete workability. Moreover, utilisation of brick powder consisting of reduced particles absorbs increased water quantity which is responsible for increased cement paste consistency and decreased slump [87,88]. The fineness of WCBP is also observed to be a reliable indicator that early days hydration of concrete could be initiated [89]. Since the creation of crystallisation nucleus that improves hydrated products propagation is associated with early days hydration of concrete, it appeared desirable to obtain WCBP of fine particles.

The surface roughness plots illustrated in Fig. 12 establish the illustrations of sample surface roughness parameters. The interpretation of findings in Fig. 12 was aided by roughness parameters presented in Table 4. From Tables 4, it is explicit that the root mean square roughness (R_q) of WCBP is slightly less than that of cement. It is reported that the presence of sulphur in specimens could occasion the increased roughness parameters [90]. It seems logical to suggest that the presence of sulphur in OPC might have probably led to the significant R_q value for OPC. Although the R_q value for WCBP is slightly less than that of OPC, this difference is marginal suggesting that the roughness of WCBP is still comparable to that of OPC. Just as with root mean square roughness values, the maximum peak to valley roughness (R_y) of WCBP is slightly less than that of OPC. It is reported that the negative values of skewness indicate that there are increased quantities of reduced troughs than peaks [91]. Unfortunately, each attempt to count these troughs and peaks was challenging as the troughs and peaks are very close to each other. Using parameters in Tables 4, it implies that WCBP has more reduced troughs than peaks compared with OPC. A fair correlation is also observed between waviness parameters of OPC and WCBP. Waviness parameters of OPC (i.e. root mean square waviness (W_q) and waviness average (W_a)) are seen to be greater than those for WCBP. Unlike the marginal differences of R_q and average roughness values existing between OPC and WCBP, the waviness parameters are seen to display increased margins.

The roughness profiles of OPC and WCBP are seen to have sharp peaks and valleys which are able to establish significant data about specimen properties. Other investigators [92], have mentioned that Kurtosis values (R_{ku}) can be related to root mean square roughness values of specimens. As such, special care has to be taken in the interpretation of R_{ku} values because of existence of mathematical relationship between the two parameters [92]. Since the R_{ku} values are mathematically related with R_q values, it was speculated that these values in Table 4, might have probably influenced the roughness parameters. A combination of roughness parameters (i.e. roughness average (R_a) and R_q), waviness parameters, skewness values (R_{sk}) and Kurtosis values suggests that the presupposition of different roughness properties between OPC and WCBP could probably be responsible for the significant influence on properties of cementitious composites. Thus, a conclusion can be drawn that WCBP in this study has significant roughness parameters that can influence the workability properties of cementitious composites.

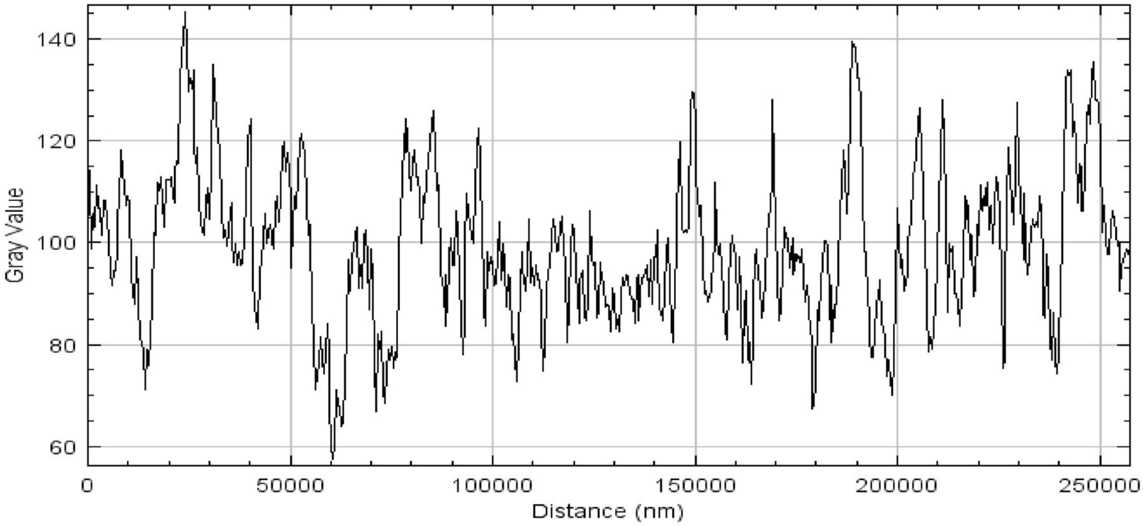
5.3.2. *Surface profile plots*

Evaluation of grain size intensity of WCBP and cement was conducted using ImageJ and this has been presented in Fig. 13. A logical evaluation was conducted to assess the grain size intensity of cement and WCBP. Gray value gives information about voids as the value for black being 0 and for white being 255. It must be mentioned that it was necessary to generate surface roughness profile plots and areas of integration of OPC and WCBP to supplement the R_q-roughness values using Gwyddion.

Both surface plots presented in Fig. 13 seem to be homogeneous and it appears that there are no significant gradients for both profiles. The profile plots provide the knowledge of grain distance and the average pixel intensity orientation [93]. There is a close agreement between computed roughness values of OPC and WCBP explained previously and their corresponding surface roughness profile plots in Fig. 13. The area of integration of the surface roughness plot of WCBP is 24853486.12945 while that of OPC is 25517265.681575. From the areas of integration findings, it is clear that OPC formulates the surface with slightly increased contact area than WCBP. Although the roughness integral value for WCBP is less than that of OPC, its roughness parameters could illustrate significant role in the behaviour of concrete with WCBP. R_q-roughness values previously presented indicate that WCBP has an R_q-roughness value slightly less than that of cement. It was thought that the inclusion of WCBP having such rough surfaces in cementitious composites could result in reduced slump of cementitious composites [87]. The reduced sizes of WCBP are also observed

Table 4
Roughness parameters of OPC and WCBP.

Specimen	R_y (nm)	R_a (nm)	R_q (nm)	W_a (nm)	W_q (nm)	R_{sk}	R_{ku}
OPC	459.181	67.112	84.595	66.852	84.868	−0.047	3.049
WCBP	432.810	66.336	82.289	47.890	59.146	−0.128	2.740



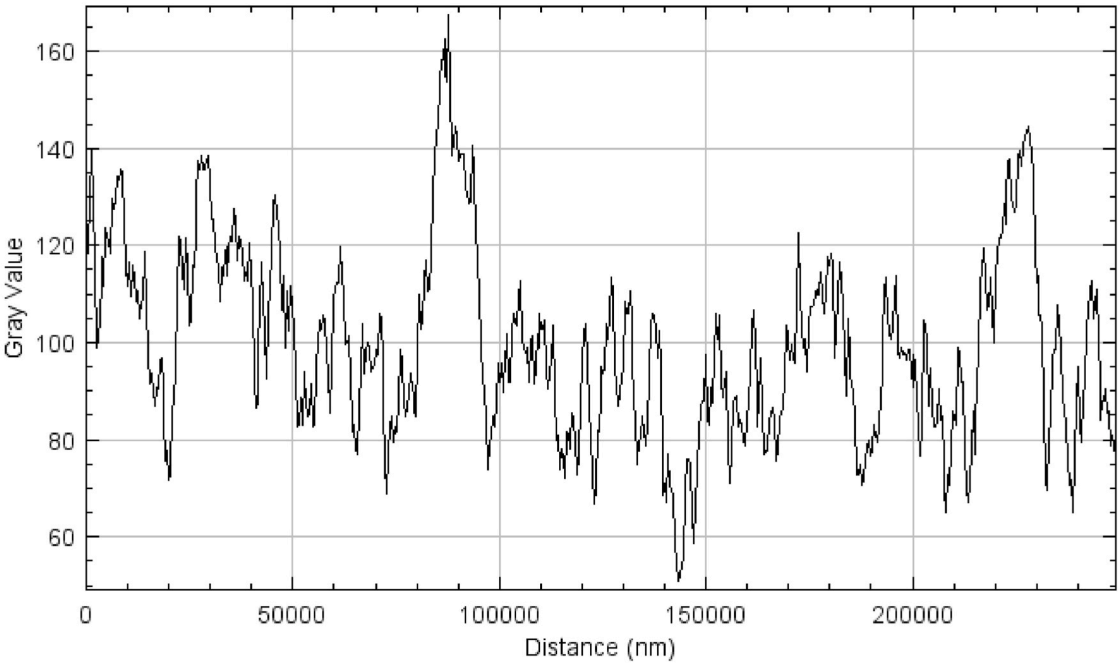


Fig. 13. The corresponding surface profile plots of OPC (top) and WCBP (bottom).

to illustrate reduced workability in contrast with bigger ones at a constant water-cement ratio [94].

5.4. Workability properties

The slump values quantified in this research ranged from 15 to 26 mm. It is known that producing concrete by inclusion of WCBP and WTR at a fixed water-cement ratio results in reduced slump of concrete [10,95]. The presence of pozzolanic materials such as WCBP in concrete places a limit in achieving improved workability of concrete due to development of increased stiffness in the mixes [96]. As explained earlier in Section 5.2, the reduction in workability due to inclusion of WCBP could be owed to the roughness characteristics of WCBP. The findings of increased consistency of cement paste containing WCBP in another study [88], lend support to this observation. Inclusion of WTR in concrete is also encountered with reduced concrete workability [10,97]. In literature, the reduction of slump due to inclusion of WTR has not only been owed to the levels of inter-particles friction occurring between tire rubber and other mix ingredients but also the general reductions in the unit weights of mixes [97–99]. In this research, there seems to be a higher probability that reduced workability was as a result of the latter cause. The decline in concrete workability behaviour attributed to inclusion of WCBP and tire rubber was compensated by adding more water to the concrete mixes. Since the workability of the specimens was maintained, the influence of physical characteristics of non-conventional concrete parameters could be analysed with greater confidence [100]. Because of the existence of friction between particles accompanied by inclusion of WCBP and WTR, additional water was advantageous in increment of workability via the particle lubrication process [101]. Also, maintaining the specific slump range is preferable in an attempt to easily compare the strengths of different concrete mixes [68]. Furthermore, although concrete with reduced slump has limitations in some applications, it can be still be used for mass dam concrete works or road pavements [102].

5.5. *Compressive strengths of modified and control concrete specimens*

Figs. 14–16 suggest that the effect of exposure to sulfuric acid was detrimental to compressive strength in comparison with samples cured in water. The most distinguishable feature of compressive strength findings was that the concrete mixes exposed to sulfuric acid exhibited decreased compressive strengths in comparison with those cured in water. For example, 30 MPa concrete grade specimens of 0P0T, 5P0T, 5P10T and 5P20T exposed to sulfuric acid had 60.69%, 58.29%, 62.28% and 74.63% reductions in compressive strengths respectively, with reference to the corresponding specimens cured using water. The reductions drawn regarding the mixes for class 30 concrete mixes should also be applicable to class 20 and 25 concrete mixtures since it is highly probable that these classes could display similar trends like that of class 30. The results of including WCBP and WTR in concrete are illustrated in Figs. 14–16. Apparently, the introduction of WTR which provided discernible reductions of compressive strength values of modified concrete mixtures compared with conventional concrete mixtures for samples cured in water, provided the same effect for specimens submerged in sulfuric acid. Moreover, the behaviour of the specimens exposed to sulfuric acid and cured in water suggests that the introduction of WTR in modified concretes resulted in decreased compressive strengths with reference to the conventional concretes.

The reductions in compressive strengths were observed at 90 days curing period for specimens submerged in sulfuric acid with reference to those cured in water. There is a possibility that moist curing might have influenced the reductions in compressive strengths of specimens. Moist curing of the samples for a period of 27 days was conducted in conformity with the standard [67]. Although moist curing was suspected to have led to reduced compressive strength values, it can be suggested that this influence was very minimal in comparison with sulfuric acid invasion. It is difficult to explain the quantitative information concerning the influence of moist curing on the specimens at the moment. It should be noted that it might appear to be misleading taking into consideration that the specimens exposed to sulfuric acid were not exposed for the entire 90 days duration. In accordance with the specifications in the code [67], moist curing of the specimens was conducted for 27 days prior to exposure to sulfuric acid. Therefore, the 90 days exposure of the specimens to sulfuric acid was not entirely appreciated. Perhaps it could be possible to ascribe these reductions in compressive strengths of specimens as partly being due to the moist curing of specimens prior to immersion in sulfuric acid. In addition, it is because of the increased H^+ ions concentration in sulfuric acid that several detrimental effects were experienced for specimens exposed to sulfuric acid. This latter cause was considered to be the major cause of these reductions. In fact, out of all mixes, never were any mixes which illustrated compressive strength losses of less than 57%. All the specimens exposed to sulfuric acid had lost more than 57% of their compressive strengths after 90 days with reference to the corresponding samples cured in water. This observation automatically placed a limitation of exposure of concrete samples to sulfuric acid. A study of data in Figs. 14–16 indicates that inclusion of 20% WTR in concrete resulted in most severe compressive strength losses for specimens submerged in sulfuric acid compared with those cured in water for all concrete classes. These findings were reliable indications of reduced bonds between rubber aggregates and cementitious composites. It seems likely that increasing the contents of WTR should lead to pronounced reductions of compressive strengths of specimens submerged in sulfuric acid compared with those cured using water. It should be remarked that no significant differences

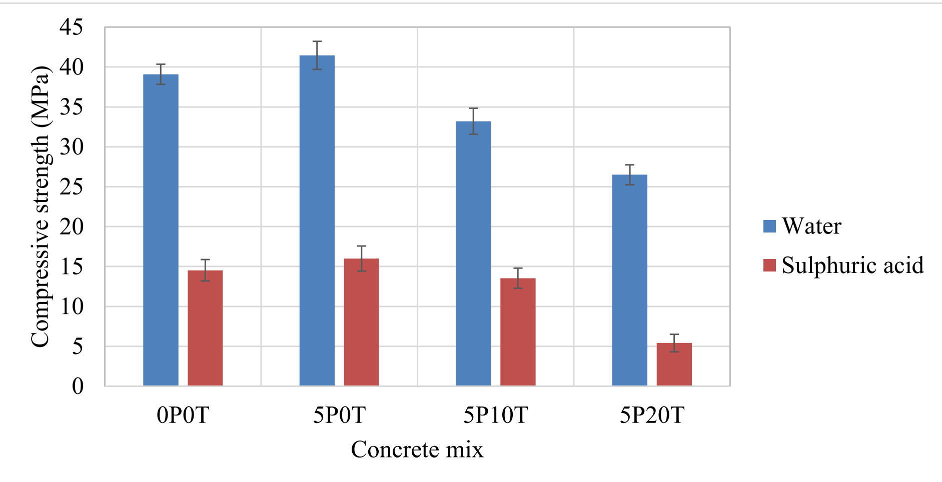


Fig. 14. Compressive strength findings for 20 MPa concrete grade mixtures submerged in sulfuric acid and cured in water.

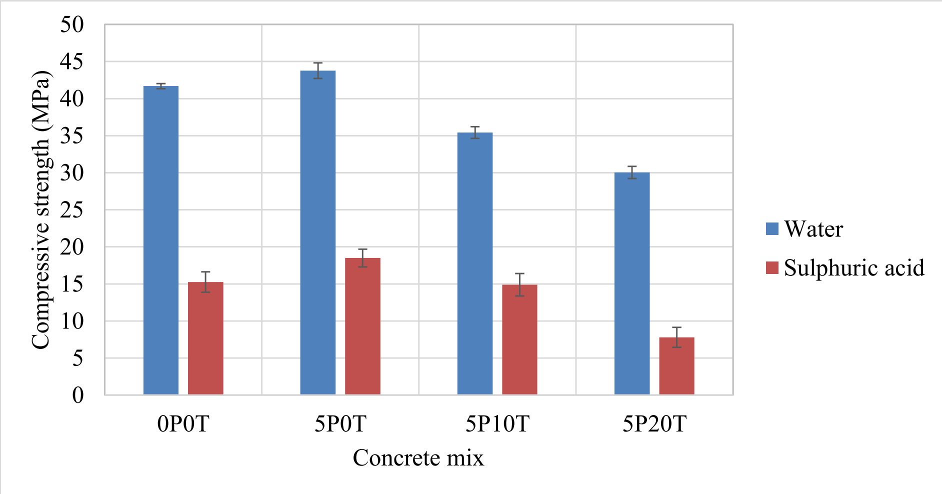


Fig. 15. Compressive strength findings for 25 MPa concrete grade mixtures submerged in sulfuric acid and cured in water.

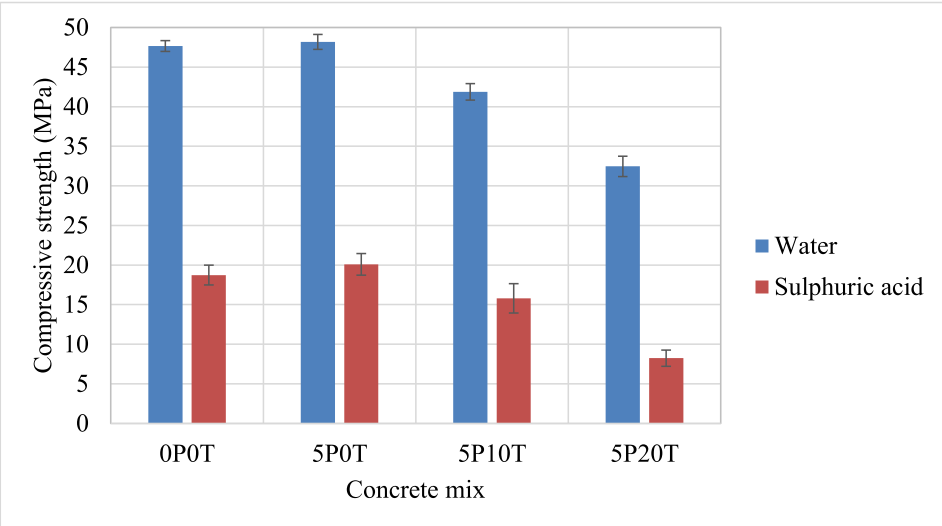


Fig. 16. Compressive strength findings for 30 MPa concrete grade mixtures submerged in sulfuric acid and cured in water.

were observed in curing temperatures between sulfuric acid and water with the range of curing temperature values determined in this research as 21.7 °C–26.5 °C. Thus, this suggested that curing temperature values were not responsible for significant distinctions in compressive strength values between the samples submerged in sulfuric acid and cured in water. Any resulting variations in temperature changes of water and sulfuric acid were considered to be minimal and were not thought to have resulted in significant influence in relation to compressive strength losses.

As mentioned previously, the losses of concrete compressive strength in specimens were owed to sulfuric acid attack resulting in weakened bonds among concrete ingredients. It is to be noted that acidic attack on cementitious composites is usually linked with the precipitations of the transformed zones which are neighbouring the sound zones [103]. According to another study [104], the replenishing sulfuric acid solution should give rise to degree of alteration of binder matrices of cementitious composites which increases with the exposure time. Following days of exposure period, significant surfaces of the specimens were altered due to exposure to sulfuric acid irrespective of the concrete mix, although the extents of alterations differed with the type of mix. The surface deteriorations generally noticed for specimens submerged in sulfuric acid in cases of self-desiccation of C–S–H gel within the transformed regions of the binder matrix [105], could be owed to chemical shrinkage. In consequence, the leaching of calcium ions might have occasioned a gradient in the ratio of calcium-to-silica within the matrix in a way that the driving force for such shrinkage might have been very pronounced around the exterior surfaces of the specimens damaged by the sulfuric acid [104]. This decalcification induced shrinkage might have probably generated differential stresses making the external surfaces of the specimens to be in tension [106]. When the tensile stresses reach the levels that induce fracture, shallow cracks could be created at the microscale level at the exterior surfaces of the specimens [104].

Minimal increments in values of compressive strength were noticed as OPC was partially replaced with 5% WCBP irrespective of concrete classes in contrast with conventional concrete mixtures for both exposure conditions. This seems to confirm the increased pozzolanic property of WCBP presented earlier. XRF and XRD findings explained previously have illustrated enhanced contents of silica and alumina and these contents were hopefully believed to maximise the pozzolanic activity of WCBP. Although perhaps coincidental, other researchers [107], have observed that the enhanced extent of pozzolanic reaction of WCBP is experienced when the curing periods range from 60 to 90 days. This is in good agreement with the conclusion drawn from the findings in this study. It is therefore clear without any reasonable doubt that minimal increments in compressive strengths of concrete mixes with WCBP irrespective of concrete grade were the consequences of improved pozzolanic reaction at increased curing period. The pozzolanic activity occasioning the creation of secondary C–S–H is responsible at reducing the large capillary pores to small pores thereby protecting concrete from ingress of harmful chemicals [108]. There seems to be a higher probability that the endurance of concrete in sulfuric acid improved due to inclusion of 5% WCBP in contrast with conventional concrete. On the other hand, the incorporation of WTR had the opposite influence on compressive strength in contrast with control concrete. There is no reason to believe that inclusion of WTR in concrete would tend to improve compressive strengths of concrete since WTR simply reduces the bonding occurring between WTR aggregate and other concrete ingredients [12,49]. Although the use of WCBP was believed to have led to increased strength for 5P0T compared with control concrete, this did not prove to be very useful when WTR was introduced. The presence of tire rubber led in decreased compressive strength in contrast with control concrete. However, it could be speculated that WCBP might have probably just reduced the strength losses in rubberised concrete mixes. Other studies [53], have pointed out that curing rubber improves the bonding between rubber and other concrete constituents. In this study, no curing of rubber was not conducted and this constitutes a limitation of this study.

5.6. Split tensile strengths for modified and control concretes

The split tensile strength findings in Figs. 17–19 give a better picture of identifiable differences of specimens submerged in sulfuric acid and specimens cured using water. At the end of 90 days, exposure of concrete to sulfuric acid had an influence on split tensile strength of concrete in same case it had on compressive strength results. For example, 20 MPa concrete grade mixes of 0P0T, 5P0T, 5P10T and 5P20T exposed to sulfuric acid revealed 35.34%, 28.19%, 31.37% and 25.07% reductions in split tensile strengths respectively, with reference to the corresponding specimens cured using water.

Data in Figs. 17–19 indicate that at 90 days, the split tensile strength values of all mixes submerged in sulfuric acid are less than those for specimens cured in water. The sources of declines in split tensile strength values of specimens submerged in sulfuric acid in comparison with those cured using water were harmful influences of sulfuric acid on concrete specimens. At the time the split tensile strength experiments were being conducted, it was thought that substantial reductions of split tensile strengths would be obtained for specimens exposed to sulfuric acid in comparison with those cured in water. Unlike compressive strength findings, split tensile strength findings seemed to indicate that the rate of split tensile strength reductions because of submerging concrete mixes in sulfuric acid was not as high as that of compressive strength results. In fact, out of all 12 mixes investigated for 20 MPa, 25 MPa and 30 MPa concrete grades, never were the split tensile strength losses of the samples larger than 43.1%. The lowest split tensile strength loss was 23.85%. The data in Figs. 17–19 illustrate that severe split tensile strength losses were never experienced with specimens containing 20% WTR and 5% WCBP for all concrete grades. This observation runs contrary to the observation of compressive strength. As previously explained, compressive strength findings illustrated that incorporation of high content of WTR (20%) in concrete was translatable to

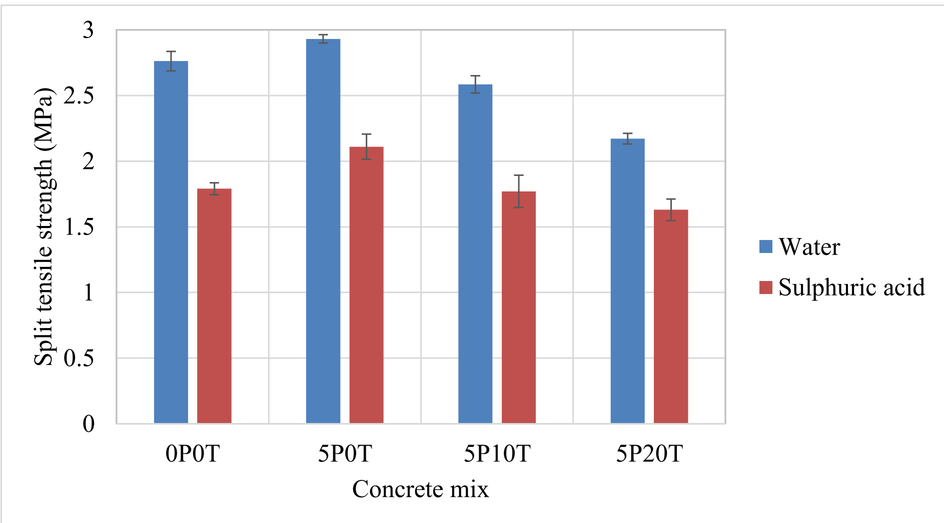


Fig. 17. Split tensile strength findings for 20 MPa concrete grade mixes submerged in sulfuric acid and cured in water.

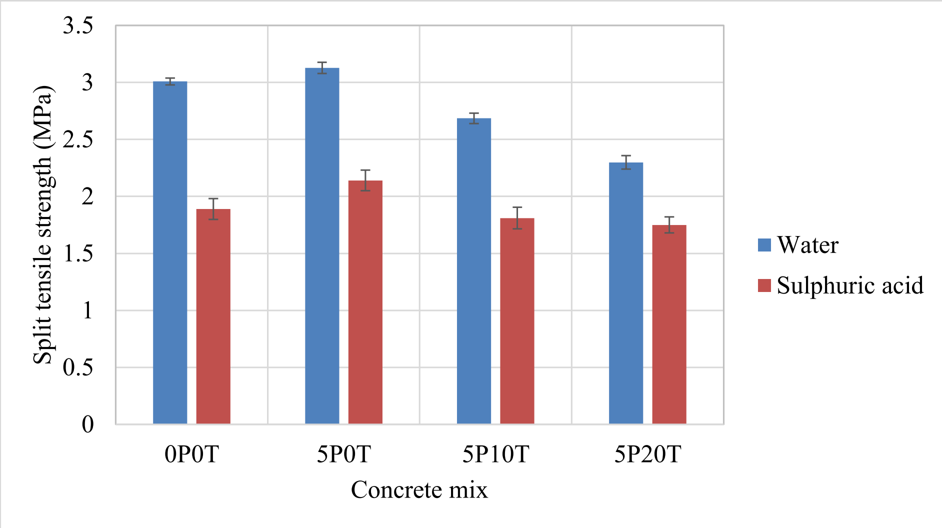


Fig. 18. Split tensile strength findings for 25 MPa concrete grade mixes submerged in sulfuric acid and cured in water.

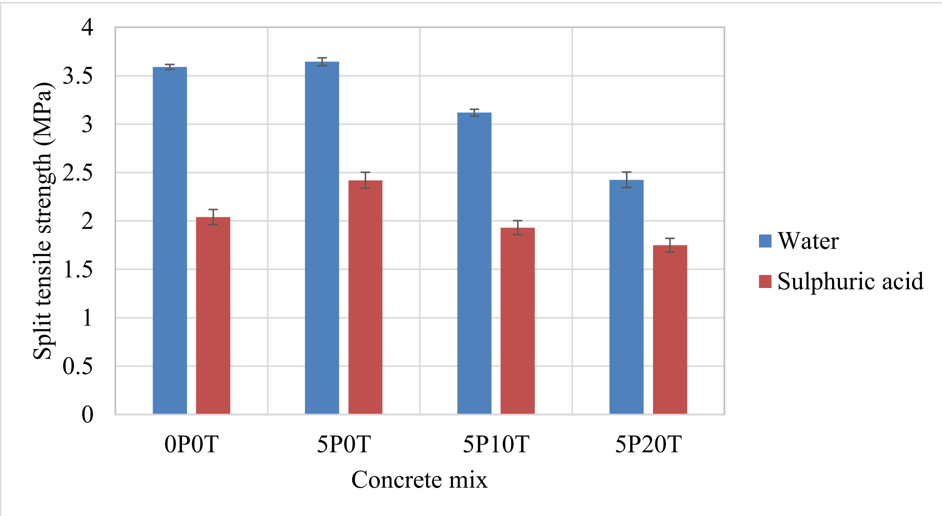


Fig. 19. Split tensile strength findings for 30 MPa concrete grade mixes submerged in sulfuric acid and cured in water.

higher compressive strength losses for samples submerged in sulfuric acid in contrast with those cured in water for all concrete grades.

At the time the analyses of split tensile strength findings were being conducted, it was reasoned that the inclusion of 20% WTR would yield the most severe split tensile strength losses due to exposure of specimens to sulfuric acid compared with curing in water. Available data do indeed show that the incorporation of 20% WTR in concrete led to the most reductions of concrete split tensile strengths compared with conventional concrete mixes, for samples submerged in sulfuric acid and the samples cured using water. However, between the exposure conditions, this high percentage of WTR was not translatable to the most split tensile strength losses for all concrete grades. This observation can be debatable and it is not clear whether this was because of the lack of direct relationships between compressive strength and split tensile strength or methodological error. Available data are seen to demonstrate the higher likelihood of the former reason being mainly the cause for these findings. By exercising greatest care and diligence during casting and testing of concrete in this study, it was hoped that success of achieving valid findings could be significantly guaranteed. In another study [109], it was noticed that no direct relationship existed between the rates of increments in compressive strength findings and split tensile strength findings. In the foregoing study, with the increments in compressive strengths of concrete specimens, the split tensile strengths also increased but at a reducing rate. Good agreement exists between split tensile strength findings in this study and those reported by researchers in the foregoing study. The findings of split tensile strength in this study seem to be justified. On the basis of exposure period, the specimens in this study were only submerged in sulfuric acid for 62 days and the findings in this study were established based on this exposure duration. Since other exposure durations were not studied in this research, it is suggested that further future research be carried out to establish the performance of concrete mixes containing tire rubber and brick powder at other exposure periods.

The incorporation of 5% WCBP was observed to enhance split tensile strength findings of modified concrete mixtures in contrast with conventional concrete mixtures exposed to either sulfuric acid or cured in water. Although there were reductions of split tensile strength for specimens submerged to sulfuric acid in contrast with those cured in water, 5P0T specimens exhibited the largest split tensile strengths among the specimens exposed to sulfuric acid. It was suspected that the existence of 5% WCBP in 5P0T specimens favoured increased pozzolanic reaction as was the case with compressive strength findings. The danger from sulfuric acid invasion seemed to have been slightly reduced due to inclusion of 5% WCBP. These observations suggest that the inclusion of WCBP tends to react with $Ca(OH)_2$ to create calcium silicate hydrates (C–S–H) especially at longer period of concrete curing. The inclusion of WCBP in concrete further suggests that the pozzolanic activity of WCBP gains significance if combined with cement, as illustrated by the sum of percentages of iron oxide, alumina and silica in XRF findings which is above 70%. It was established by XRF test results that the sum of percentages of alumina, silica and iron oxide in WCBP was 85.93% and such amount of silica was as a consequence of thermal history of clay bricks. The significance of these increased contents of iron oxide, alumina and silica in WCBP common to more pozzolanic materials is fairly a good measure of enhanced pozzolanic reactivity leading to increased compressive strengths. The increased split tensile strength of concrete containing 5% WCBP compared with conventional concrete show evidence of silica going into reactivity with $Ca(OH)_2$ to create calcium silicate hydrates (C–S–H). This is in complete agreement with the XRF and XRD findings previously discussed and such findings can be relied upon. The reductions in split tensile strength were more evident in concrete specimens when WTR aggregates were incorporated in contrast with conventional concrete specimens. The higher the content of WTR in concrete, the greater the rate of reduction of the split tensile strength. It is worth pointing out that inclusion of WTR in concrete hardly ever leads to improved strength findings in contrast with normal concrete. This is mainly because of the simultaneously occurring variables of segregation of surfaces between WTR and cementitious paste occurring during crack expansions [110] and reduced bonding between cementitious paste and WTR [111].

5.7. Mass losses and changes in diameters of samples

The relationship of mass losses of different mixtures submerged in sulfuric acid and cured in water is depicted in Table 5. Table 5 also enlists the data of diameter changes for all the mixes. The diameter changes were generated by obtaining the differences between diameters measured before and after immersion in sulfuric acid and curing in water. In general, the changes in diameters were only experienced for the specimens exposed to sulfuric acid. From samples cured in water, out of all the mixes, never were any mixes that experienced diameter changes. In Table 5, the mass losses of samples exposed to sulfuric acid increased in comparison with those cured in water. There appeared to be no proof that curing of concrete specimens in water affected the mass losses of the samples. This was illustrated by lack of significant findings on mass losses of the samples. Neither WCBP nor careful casting of concrete were believed to have resulted in significant mass losses for specimens cured in water. These findings suggest that in spite inclusion of WTR and WCBP in concrete, this was not translatable to any significant mass losses following curing in water. It can be suggested that this lack of significant trend can probably be explained by little or no harmful substances associated with curing in water which could have resulted in no significant masses losses.

From the findings of mass losses of 5P10T and 5P20T, it is clear that the density of concrete, like the compressive strength and split tensile strength, is the inverse function of the quantity of the WTR aggregate (with the quantities of WCBP for the two mixes being equal). Other investigators have also attempted to investigate the effect of WTR on concrete density and have observed that inclusion of WTR aggregates reduces the concrete densities [112]. It is reasonable to indicate that the enhanced mass losses of rubberised concrete mixtures containing WCBP were attributed to enhanced concrete degradation from sulphate attack. It must be remembered that the compacted bulk densities of non-conventional concrete materials are of significance in concrete production but only when these bulk densities vary significantly in contrast with those of control concrete materials. It is once again established that these findings are reliable justifications of reduced bonding between rubber aggregates and cementitious composites. In the entire study, the concrete samples exposure to sulfuric acid did not seem to illustrate any beneficial effect compared with curing in water. The suspicion arose immediately that since sulfuric acid attacked calcium hydroxide forming calcium sulphate [113], this resulted in the mass losses in concrete specimens. There is every likelihood that there was damage of fragile silica gel that is created after reactivity with calcium silicate hydrate and this to a certain extent leads in concrete failure, cracks, strength loss and concrete expansion [113].

According to the findings, the inclusion of 20% WTR in concrete illustrated the most diameter losses compared with other mixes for specimens exposed to sulfuric acid. It must be mentioned that no identifiable changes in diameters were noticed on cylinder specimens cured in water. There was no proof that the cylindrical diameters of the specimens decreased or increased following curing in water. It

Table 5
Diameter changes and mass losses of specimens exposed to sulfuric acid.

Mixture	Class 20		Class 25		Class 30	
	Mass loss (g)	Diameter loss (mm)	Mass loss (g)	Diameter loss (mm)	Mass loss (g)	Diameter loss (mm)
0P0T	333.33	1.67	313.50	1.67	323.00	1.67
5P0T	286.83	1.33	251.83	1.33	216.83	1.67
5P10T	469.00	3.33	416.33	3.00	434.67	2.67
5P20T	511.50	4.00	471.17	3.67	448.93	3.33

was reasoned that curing in water was not translatable to any significant changes in diameter. The maximum loss of diameter change was observed with 5P20T concrete mix for 20 MPa concrete grade following exposure to sulfuric acid. It can be suggested that this was because of reduced concrete grade. Since OPC acts as a thin filler, there is an increment in compactness of concrete because of increment in OPC content [114]. One of the most interesting findings was that inclusion of 5% WCBP in 5P0T mixes seemed to illustrate the most reduced diameter losses for all concrete grades. It can be suggested that the pore refinement resulting in creation of additional hydration products and filler effect of WCBP might have probably protected concrete from diameter losses [115].

The diameter and weight losses of concrete samples submerged in sulfuric acid are because of leaching of calcium sulphate and this is created in case of invasion of sulfuric acid on calcium hydroxide [113]. There are destructions of fragile gels which are created after the reactions with calcium silicate hydrates (C–H–S). In addition, from the initial reaction, calcium sulphate is noticed to participate in reactions with cement to form the phase of calcium aluminate. In consequence, these observations initiate concrete disintegration and strength losses of concrete [113].

5.8. Visual inspection

The cube and cylindrical specimens tested after 90 days of curing using water and submerging in sulfuric acid are depicted in Figs. 20 and 21. In this study, the characteristics of the surfaces of the samples were carefully evaluated to explore the influence of exposure to sulfuric acid and curing in water on performance of rubberised concrete containing WCBP. Further inspection of the specimens involved scratching of the surfaces of the samples and attempts to remove WTR aggregates and coarse aggregates from the specimens. It should be noted that not all specimens have been presented but only representative samples are depicted in Figs. 20 and 21. From the figures, it is clear that the samples cured in water experienced no concrete disintegration. In contrast, the samples exposed to sulfuric acid heavily suffered from concrete disintegration. Subsequently, spalling and cracking (subject to the experimental considerations) were propagating through the specimen surfaces. It was difficult to estimate the exact quantities of the spalled materials; however qualitatively, it seemed that an increasing trend was observed when the increased quantities of tire rubber were incorporated.

From Figs. 20 and 21, it is clear that exposure to sulfuric acid led to concrete disintegration. No concrete disintegrations were

Fig. 20. Cubes and broken cylinder specimens for class 30 (5P0T) concrete mixes after testing: cured using water (left column) and exposed to sulfuric acid (right column).

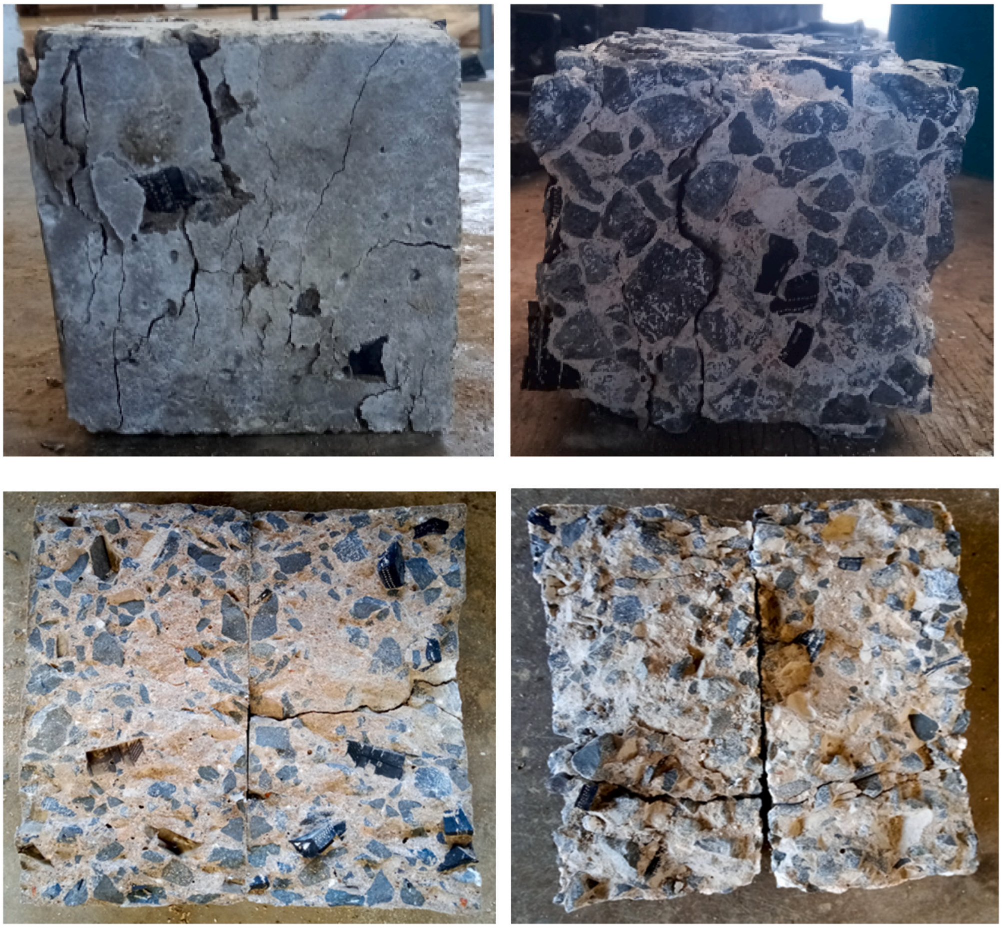


Fig. 21. Cubes and broken cylinder specimens for class 25 (5P10T) concrete mixes after testing: cured using water (left column) and exposed to sulfuric acid (right column).

observed for concrete specimens cured in water. The pictures in Figs. 20 and 21 illustrate that although the rubber content was much less than coarse aggregates, it contributed to reductions of compressive and split tensile strengths in contrast with control concrete. It is because of decline of bonding between WTR aggregates and other concrete constituents. The fact that surfaces of tire rubber aggregates in Fig. 21 are clearly visible is a reflection of limited bonding between tire rubber and cementitious composites. Another proof was observed immediately after breaking the cylinder specimens as tire rubber aggregates seemed to be easily removed in contrast with coarse aggregates. This observation as already mentioned reduced the probability of observing improved compressive strengths and split tensile strengths in contrast with conventional concrete. The reaction between sulphate ions from the sulfuric acid and calcium hydroxide leads in the formations of gypsum. The created gypsum participates in a reactivity with calcium aluminates to create ettringite in cement matrix [104].

From the specimens in Figs. 20 and 21, it is clear that depositions of flaky or white substances are noticed on the outer layers of specimens submerged in sulfuric acid. The outer layers of the samples were very soft in such a way that scratching them with fingernails caused substantial damage to the layers. Following breaking of the cylinder specimens, the flaky or white deposits were also observed in the internal zones of the samples submerged in sulfuric acid. The study by other authors [23], showed that cement mortar had deposits of flaky or white substances when exposed to sulfuric acid. This present study includes the breaking of the specimens to verify the existence of flaky or white substances in the internal zones of the specimens, which was ignored by the foregoing study. From the figures, it seems that disintegration of the specimens accelerated when tire rubber aggregates were incorporated. Since brick powder was included in the rubberised concrete, it could be speculated that this inclusion of WCBP might have probably reduced the rate of degradation of rubberised concrete specimens. There is a possibility that inclusion of WCBP might have improved the bonding by arresting the compressive strength and mass losses from acid invasion. It is known that compressive strength and mass losses are arrested due to inclusion of appropriate contents of pozzolanic materials substituting cement in concrete [51,52]. This is attributed to enhanced micro-aggregate fillings and pozzolanic activity effects which are largely experienced at increased curing periods and these obstruct the attack of suphuric acid in concrete [51,87].

6. Conclusions

Through this experimental work, some important conclusions were established.

1. The concrete specimens' exposure to sulfuric acid resulted in split tensile strength, compressive strength, mass and diameter losses of concrete mixes compared with curing in water.
2. The compressive strength findings showed that all the specimens exposed to sulfuric acid had lost more than 57% of their compressive strengths after 90 days with reference to the corresponding specimens cured in water. In contrast, out of all the mixes investigated for all concrete grades, never were the split tensile strength losses of the samples submerged in sulfuric acid greater than 43.1% compared with those cured in water.
3. Concrete specimens containing 5% WCBP showed enhanced compressive strengths and split tensile strengths in contrast with conventional concrete specimens. Some possible explanations of this behaviour could be the increased pozzolanic activity of WCBP linked with increased curing periods. The inclusion of 5% WCBP was suggested to result in decreased rate of compressive and split tensile strength declines within rubberised concrete.
4. The outcomes of incorporation of WCBP and WTR in concrete were examined. The latter resulted in reduction of concrete performance particularly under sulfuric acid attack compared with control concrete. The former was believed to give rise to reduced rate of concrete disintegration due to sulfuric acid invasion.
5. No significant mass and diameter losses were noticed for specimens cured in water. Some possible explanations of significant existence of harmful substances in water resulting in mass and diameter losses of concrete specimens were ruled out.
6. Depositions of flaky or white substances were noticed on the outer layers of specimens submerged in sulfuric acid. The outer layers of the samples were very soft in such a way that scratching them with fingernails caused substantial damage to the layers.
7. The present study illustrates the findings obtained following 90 days tests for samples submerged in sulfuric acid and cured in water. Further future studies are recommended to capture the behaviour of concrete mixes containing tire rubber and brick powder at longer exposure and curing durations.

Author contribution statement

David Sinkhonde: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Richard Ocharo Onchiri; Walter Odhiambo Oyawa; John Nyiro Mwero: Conceived and designed the experiments; Analyzed and interpreted the data.

Data availability statement

Data will be made available on request.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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