

# SORBTIA BETONULUI CONȚINÂND REZIDURI DE MINEREU DE FIER SORPTIVITY OF CONCRETE CONTAINING IRON ORE TAILINGS

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*Sustainability of the construction industry is a challenge to every concerned civil Engineering professional. Iron ore tailings is an industrial waste derived from the production process of iron ore. This study consider the possibility of using this waste for making concrete. The tailings was combined with sand in varying proportion as fine aggregate in concrete. One control sample with no tailings and four others incorporating tailings at varying percentage as partial replacement for sand were used for the experimental program. The physical and microscopic properties of the tailings were compared with those of sand. The mechanical properties and microstructure of the hardened concrete were also studied. The outcomes of experimental tests shows that the tailings increased the denseness of concrete matrix and improved the water resistance of the concrete.*

**Keywords:** Fine aggregate; Concrete; Iron ore Tailings; Strength; Microstructure; Water sorptivity.

## 1. Introduction

The sorptivity of concrete refers to the measurement of the rate of absorption of water by capillary suction of unsaturated concrete which is placed in contact with water. The determination of sorptivity of concrete has been found to be very useful as a way of measuring the ability of concrete to absorb and transmit water by capillarity. Studies have been conducted on the sorptivity of concrete made with natural aggregate, meanwhile there is need to evaluate the sorptivity of concrete containing iron ore tailings as fine aggregate, partially replacing sand. The major concern in the assessment of all absorption tests is the determination of the initial moisture condition, which also affects sorptivity measurements. In poorly compacted concrete, where entrapped air voids do not get filled with water, it is possible the absorption may be lower compared to a thoroughly compacted concrete sample. However, due to the difficulties associated with the absorption tests, and because permeability test measures the response of concrete to pressure [1], a phenomenon which is not common with concrete in its natural state, the sorptivity test has been found to be a better option of assessing the quality of concrete. The fluid penetrability of concrete is described in the literature in varying terms, it is therefore necessary to present the relevant mathematical expressions that explains the phenomenon of water sorptivity. This is briefly discussed under experimental program in this study.

According to United State Geological Survey, the world annual iron ore production for the year 2015 is 3320 million metric tonnes [2]. Abundance of tailings waste are therefore generated from processing of the ore, since on the average only 17 – 25 % of the iron ore are extracted from the main raw material magnetite and haemetite[3]. In addition, almost every mineral producing country is facing the problem of better utilization of mine waste because of its accumulation and lack of suitable storage space. Increasing public awareness about various health hazards and stringent contamination norms of pollution monitoring authorities have also created pressure on governments as well as the private sector to devise waste management and utilization solutions to prevent various environmental hazards.

The properties of tailings waste can not be generalized. The composition of iron ore tailings depends directly on the composition of the raw ore (magnetite or haemetite, or combination of both) and the process of mineral extraction used on the ore [4]. Due to this varying properties of the tailings the best option of evaluating its suitability is to subject it to laboratory testing. In some previous research work, iron ore tailings was used as fine aggregate to produce ultra-high performance concrete [5], fine particles less than 75 $\mu$ m in iron ore tailings sand were found to be beneficial to the reduction of expansion induced by alkali-silica reaction (ASR) [6]. The waste obtained from hematite, was also used to improve concrete resistance to abrasion and

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surface frictional properties [7]. Various means by which the properties of concrete can be improved have been reported by previous research. Some forms of mineral binders were used to reduce pores and permeability of concrete [8, 9], different forms of sand in concrete as siliceous material to produce concrete with high resistance to water [10 - 12].

In Kota Tinggi, Johor, Malaysia, at ZCM Mines, abundance of tailings are generated from the production process of iron ore. Presently the local populace and the company use the tailings for some forms of construction outside and within the mine area. This research is tailored towards establishing a more beneficial means of using this industrial waste in concrete.

## 2. Description of materials and method of work

### 2.1. Materials

The fine aggregate used for concrete production consists of iron ore tailings and natural sand. The tailings was obtained from a mineral mine located in

Kota Tinggi while the sand was collected from a local quarry. The photographic image of the tailings compared with sand is shown in Figure 1. Particle size distribution of sand and iron ore tailings, including that of coarse aggregate is represented by the logarithmic scale graph shown in Figure 2. The materials were used under saturated surface dry condition. The physical properties of fine aggregate determined experimentally are shown in Table 1. X-ray fluorescence (XRF) test was also conducted on the iron ore tailings for the purpose of knowing the chemical composition of the tailings as indicated in Table 2. The FESEM morphology of natural sand at magnification of 2mm compared with that of iron ore tailings at the same magnification is shown in Figure 3. 10mm crushed granite was used as coarse aggregate for the production of concrete samples. The ordinary Portland cement brand, with strength class of 42.5 in accordance with the British standard [13] was used as binder for preparing the concrete samples. The hydration process was enabled using portable water obtained in the laboratory.



Fig. 1 - Photographic Image of Sand Compared with Iron ore Tailings

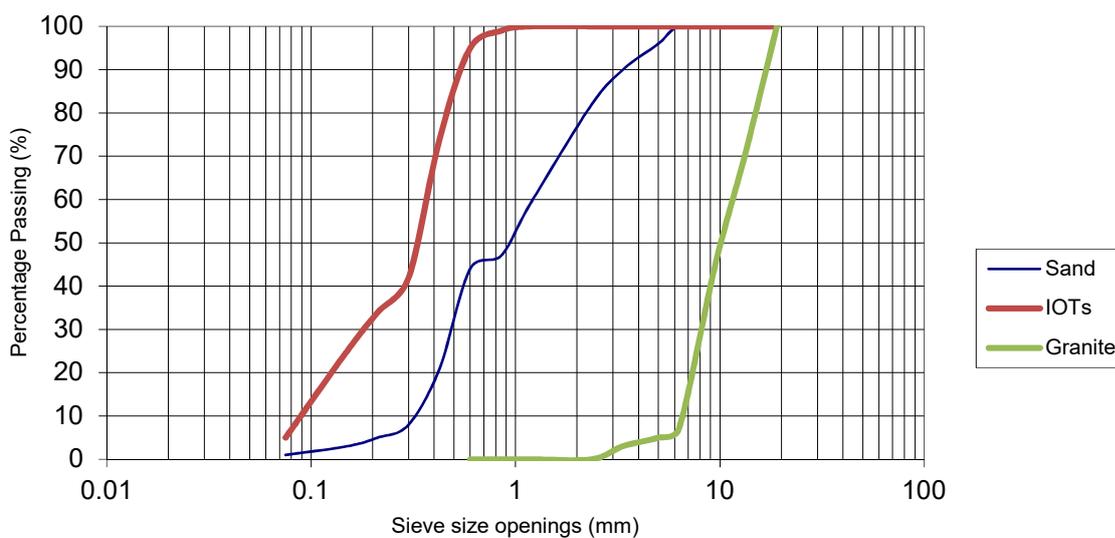


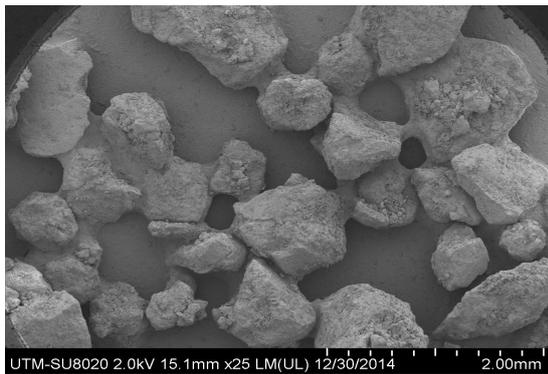
Fig. 2 - Grain size distribution of aggregates

Table 1

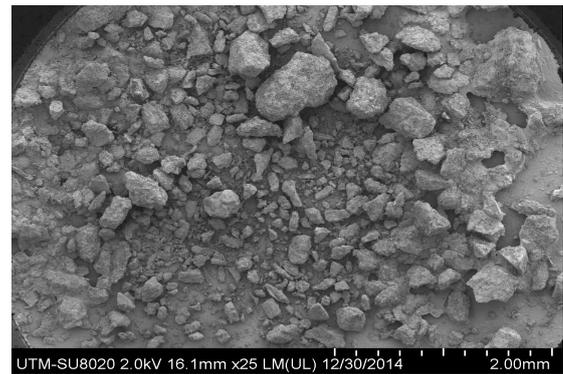
The physical properties of fine aggregate		
Physical properties	Fine aggregate	
	Natural sand	Iron ore tailings
Size Passing 600µm %	44	95
Coefficient of uniformity	3.7	4.7
Coefficient of curvature	0.02	0.01
Porosity %	14	12.1
Specific gravity	2.65	2.91
Fineness Modulus	3.2	1.4
Loose unit weight kg/m <sup>3</sup>	1459	1598
Compacted unit weight kg/m <sup>3</sup>	1696	1817

Table 2

Oxide Compositions of Iron Ore Tailings														
Chemical content	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	K <sub>2</sub> O	MnO	MgO	Na <sub>2</sub> O	TiO <sub>2</sub>	SO <sub>3</sub>	SnO <sub>2</sub>	ZnO	As <sub>2</sub> O <sub>3</sub>	PbO
% Composition	37.2	22.1	10.7	8.52	1.71	1.04	0.96	0.46	0.43	0.26	0.22	0.21	0.17	0.16



Sand



Iron ore Tailings

Fig. 3 – FESEM Morphology of Sand compared with Iron ore Tailings

## 2.2. Methods

The methodology of research includes theoretical knowledge of the phenomenon of sorptivity, design and proportioning of concrete mix, determination of fresh and hardened properties of concrete.

### 2.2.1. Sorptivity of concrete

The penetrability of concrete is described in the literature in varying terms, it is therefore necessary to present the relevant mathematical expressions that explains the phenomenon of water sorptivity as follows:

$$i = St^{0.5} \quad (\text{Eqn. 1})$$

Where,

$i$  = increase in mass since the beginning of the testing per unit of cross-sectional area in contact with water, divided by the density of water. Working in metric units,  $i$  can be expressed in mm.  
 $t$  = time, measured in minutes, at which the mass is determined.

$S$  = sorptivity in mm/min<sup>0.5</sup>

The sorptivity of the concrete samples was determined based on guidelines in ASTM C1585-13 [14]. The test measures the increase in the mass of a specimen resulting from absorption of water as a function of time in which only one surface of the specimen is exposed to water. For each type of concrete, three specimens 500 x 100 x 100 mm concrete prisms were cured in water at laboratory condition for 28 days. The measurement of sorptivity

was done at laboratory environment under standard relative humidity to induce a consistent moisture condition in the capillary pore system. The lower part of the prism measuring 5mm from the nethermost was submerged in water and water ingress of unsaturated concrete was dominated by capillary suction during initial contact with water. The rate of ingress of water by absorption due to capillary rise was calculated using the average result of three prisms specimen for all the concrete types.

### 2.2.2. Design of normal strength concrete

The procedure in the British method for the design of normal strength concrete made with ordinary Portland cement [13] was followed for the design. The mix design ensures proper selection and proportioning of materials to produce concrete of the required properties. Normal strength concrete was designed to have 28 day characteristics concrete cube compressive strength of 30 N/mm<sup>2</sup>, Target mean strength of 43 N/mm<sup>2</sup>, based on assumed Slump of 100 mm, crushed aggregate with maximum size of 10 mm and specific gravity of 2.7 using water-cement ratio of 0.54.

The output of design specify 250 Kg/m<sup>3</sup> water content, 463 Kg/m<sup>3</sup> quantity of cement, 769 Kg/m<sup>3</sup> fine aggregate, 868 Kg/m<sup>3</sup> Coarse aggregate. The fine aggregate content were further proportioned between sand and iron ore tailings to produce five different types of concrete samples.

Table 3

Types of concrete and their composition						
Concrete Type	Description	Composition of concrete Kg/m <sup>3</sup>				
		Water	Cement	Granite	Sand	IOTs
CTO	Concrete with 0% IOTs	250	473	868	769	0
CZT10	Concrete with 10% IOTs	250	473	868	692	77
CZT20	Concrete with 20% IOTs	250	473	868	615	154
CZT30	Concrete with 30% IOTs	250	473	868	538	231
CZT40	Concrete with 40% IOTs	250	473	868	461	308

Table 4

Workability	Concrete sample				
	CT0	CT10	CT20	CT30	CT40
Slump (mm)	81	79	67	59	53
Compacting factor	0.92	0.91	0.90	0.90	0.89

The natural sand was partially replaced with iron ore tailings at replacement level of (0, 10, 20, 30 and 40) %. Table 3 shows the five different types of concrete samples produced and the details of the concrete mix proportioning of materials.

### 2.2.3. Testing of fresh and hardened concrete

Slump test [15] and compacting factor [16] test were conducted on the fresh concrete. Samples of hardened concrete cubes prepared, were also tested for ultrasonic pulse velocity [17], compressive strength [18], flexural strength [18], density [19] and sorptivity [14]. The field emission scanning electron microscopy (FESEM) of the samples were also studied. The testing of samples was done based on guidelines given in the relevant British standard and the American standard for testing materials as reflected in the reference.

## 3. Results and Discussions

### 3.1 Workability of concrete

The concrete slump test is an empirical test that measures the workability of fresh concrete. The test determines the consistency of the fresh concrete samples. The control sample had the highest value of 81mm slump while the concrete sample with 40% iron ore tailings as fine aggregate had the least value of 53mm. Due to the high affinity of iron ore tailings for water at the fresh state of

concrete, there was reduction in slump values for fresh concrete samples containing IOTs.

The compacting factor test is another useful tool used to measure the workability of fresh concrete. There was no significance difference in the values obtained for the control sample and those containing iron ore tailings. The compacting factor for the reference sample gave 0.92 and for those samples containing iron ore tailings, the factor ranges between 0.89 - 0.91. Higher content of IOTs results in greater values of compacted weight. The BS 1881-103 [16] prescribes for normal strength concrete, compacting factor value range of 0.8 to 0.92. Table 4 shows the results of the workability tests.

### 3.2 Density and ultrasonic pulse velocity (UPV)

In order to determine the uniformity of concrete with respect to the presence of cracks or voids and changes in properties of concrete with time, the pulse velocity of the concrete samples was measured. The ultrasonic pulse velocity determination is a convention because the path length over which the pulse travels may not strictly be known. Density of materials is also an important physical property that influence pulse velocity. Correspondingly the density of materials has effect on the density of concrete. The density of concrete samples and the ultrasonic pulse velocity as related to iron ore tailings content are shown in Figure 4 and Figure 5 respectively.

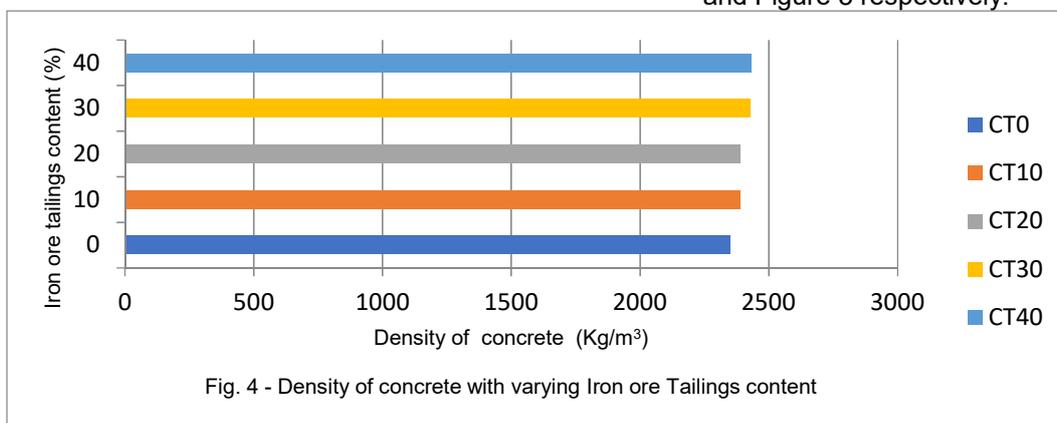
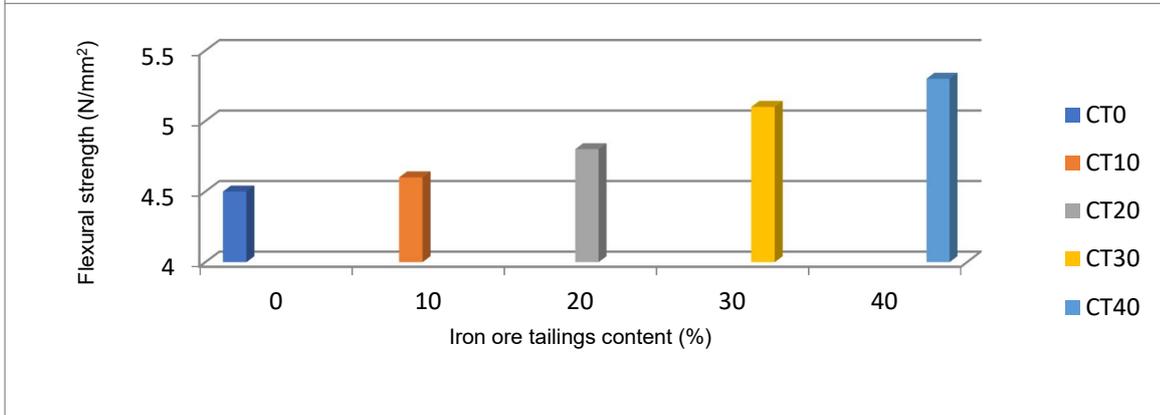
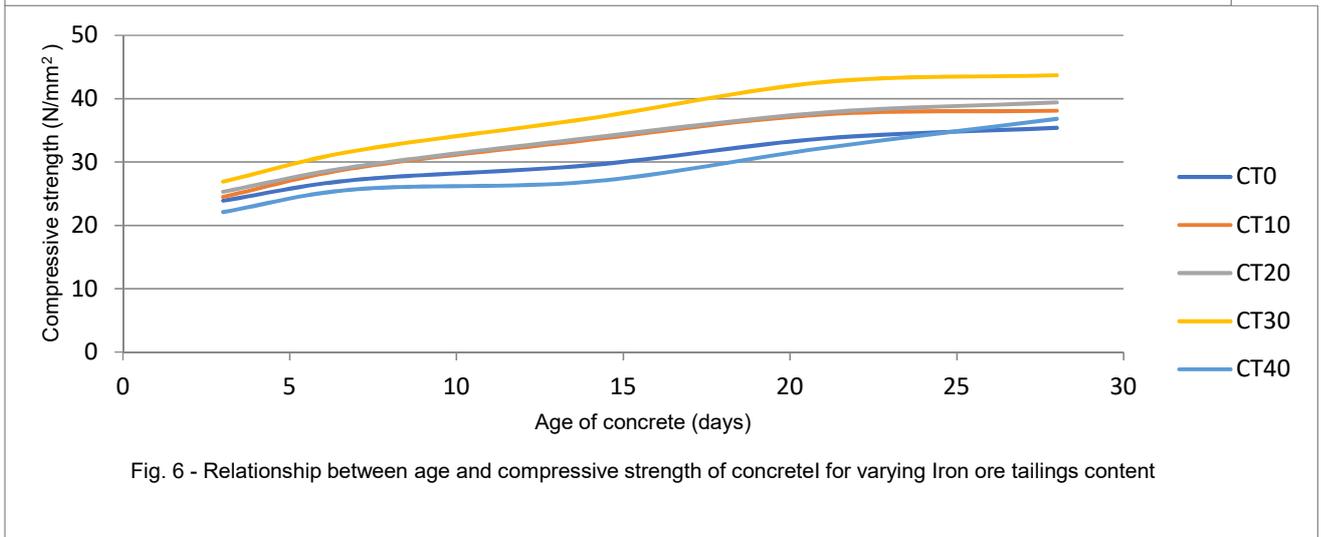
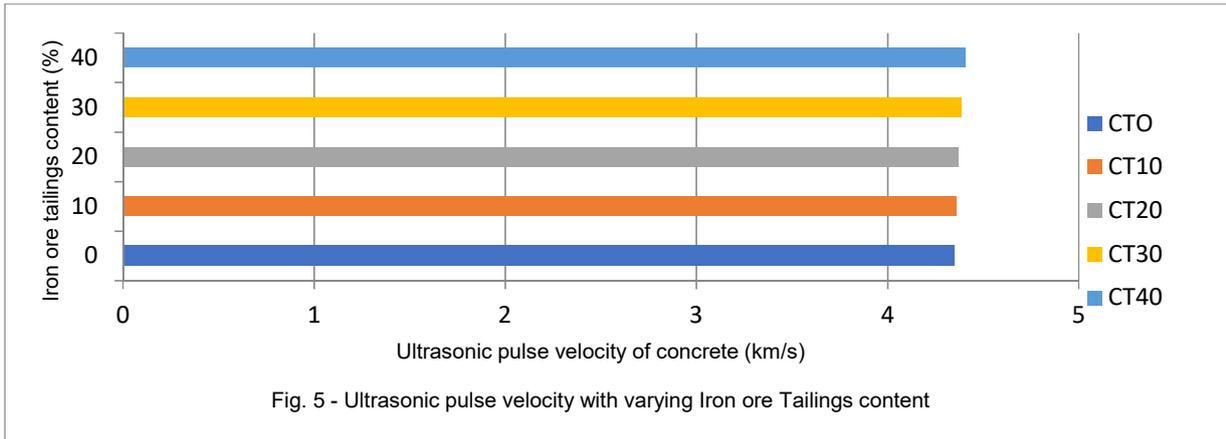


Fig. 4 - Density of concrete with varying Iron ore Tailings content



**3.3. Compressive strength**

The compressive strength is the most important properties of hardened concrete. It's the property generally specified in construction design and quality control [20]. All concrete samples with partial replacement of sand with iron ore tailings as fine aggregate gave higher values of compressive strength than the reference sample. The relationship of age of concrete with the compressive strength for all the concrete samples is reflected in Figure 6.

**3.4. Flexural strength**

The flexural strength of concrete is usually lower than that of corresponding mortar [1] implying that the finer content of the matrix contributes more in improving the flexural strength. The IOTs used as fine aggregate can be considered to be responsible for higher flexural strength recorded by concrete samples containing iron ore tailings. The flexural strength is more sensitive to features and defects in the microstructure such as micro-cracks in the material, than the compressive strength [21, 22]. The flexural strength of concrete prism samples as related to iron ore tailings content is shown in Figure 7.

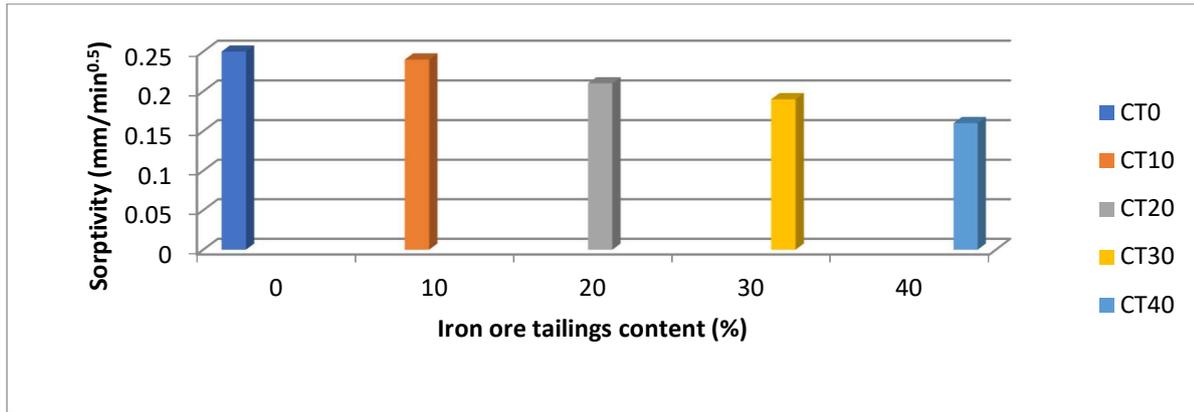


Fig. 8 - Water Sorptivity of concrete with varying Iron ore tailings content.

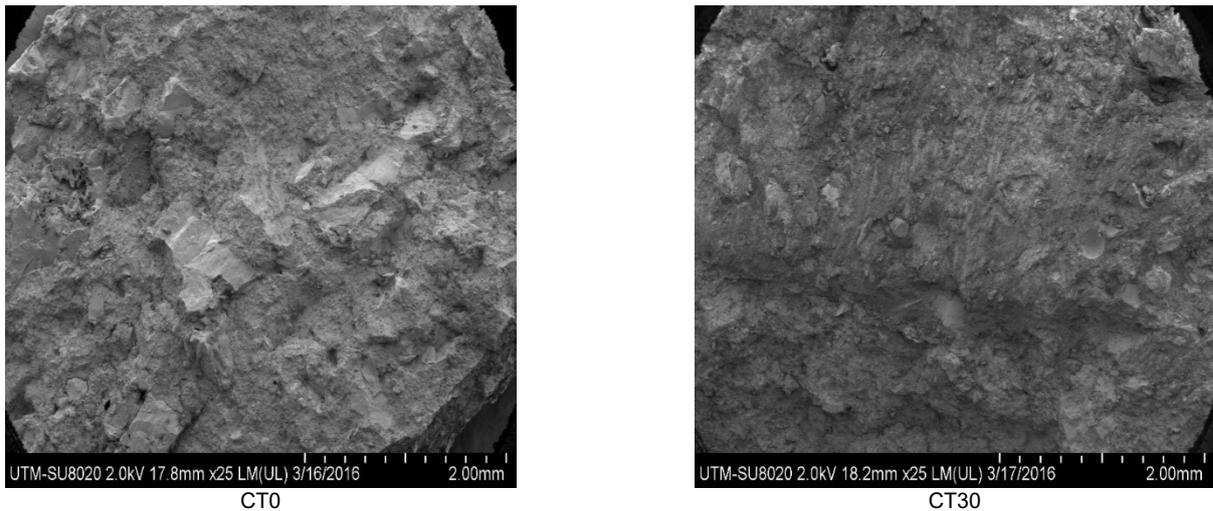


Fig. 9 - FESEM morphology of Iron ore tailings concrete (CT30) compared with reference concrete (CT0).

### 3.5. Sorptivity test

Sorptivity test measures the rate of absorption of water by capillary suction of unsaturated concrete placed in contact with water in such a way that no head of water exists [1]. It has been found to be a precise quantity that could be measured rapidly and with repeatable accurate results. It's also sensitive to the quality of the outer zone of concrete members and has proven effective in revealing poor placing and finishing techniques in concrete [23]. The iron ore tailings concrete type CT40 recorded the least value of sorptivity  $0.16 \text{ mm/min}^{0.5}$  while concrete type CT0 gave  $0.25 \text{ mm/min}^{0.5}$ . This reveals that increasing the content of iron ore tailings as fine aggregate in concrete results in lower values of sorptivity. It therefore implies that the rate of absorption of water by capillary suction of unsaturated concrete decreases with the addition of iron ore tailings. The sorptivity of concrete in relationship with the content of iron ore tailings is shown in Figure 8

### 3.6. Field emission scanning electron microscopy (FESEM)

The Field emission scanning electron microscopy (FESEM) provides the morphology of concrete samples containing iron ore tailings

compared with the reference sample. The electrons are produced by a field emission source in the microscope. The sample material was scanned in a random pattern by an electron beam. The FESEM morphology of the reference sample CT0 at magnification scale of 2 mm in contrast to the sample that contains 30 percent iron ore tailings CT30 is shown in Figure 9. The FESEM morphology reveals that more pore space can be seen in the concrete sample CT0 compared to CT30 implying that the iron ore tailings was able to improve the packing density of the concrete. Also the interfacial zone between the aggregate and the cement paste looks porous and less resistant than the cement matrix in the control sample. The C-S-H hydrates which are bound weakly to the aggregate can easily be torn. The fracture surface of CT0 can be considered as intergranular. In the case of CT30, there is intimate bond between the aggregate interface and the cement paste. There is no transition zone and no feasible cracks around the aggregate. The fracture surface can be considered as transgranular.

### 3.7. Correlation between sorptivity and flexural strength

Flexural strength of iron ore tailings concrete is

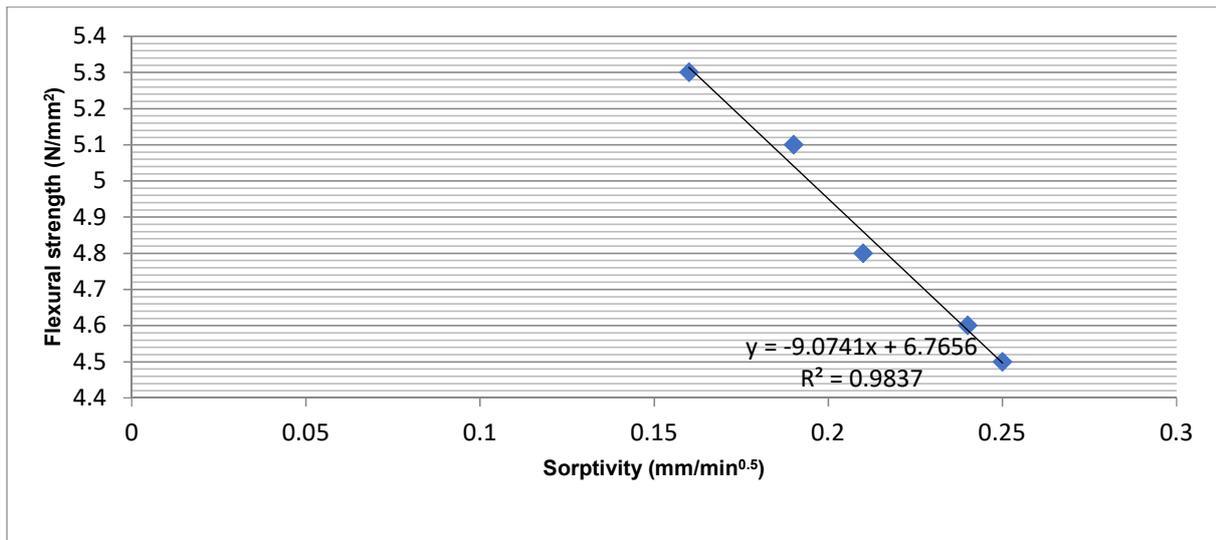


Fig. 10 - Relationship between sorptivity and flexural strength of concrete after 28 days of standard curing.

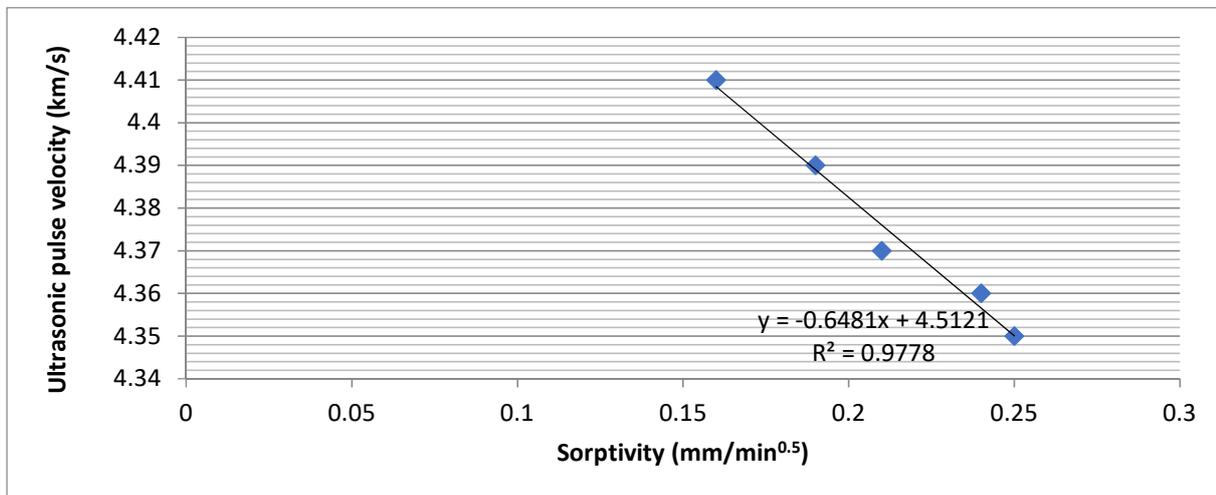


Fig. 11 - Relationship between sorptivity and ultrasonic pulse velocity of concrete after 28 days of standard curing.

closely related to the sorptivity characteristics. The flexural strength of the concrete is a good indication of existence of strong bond between the aggregates with the cement paste. This strong bond is also responsible for the concrete water sorptivity performance. The relationship between the water sorptivity and flexural strength is shown in Fig. 10.

### 3.8. Correlation between sorptivity and ultrasonic pulse velocity

The correlation between the water sorptivity and ultrasonic pulse velocity is plotted in Figure 11. It can be observed that there is strong inversely proportional relationship between water sorptivity and ultrasonic pulse velocity of iron ore tailings concrete. These results suggest that the small particle size of iron ore tailings was able to fill the pore space in concrete thereby producing more dense concrete, which gave higher values of ultrasonic pulse velocity and reduced the rate of water capillary by suction.

### 3.9. Durability of iron ore tailings concrete

It's required that every concrete structure must be able to withstand the processes of wear to which it is exposed, without significant deterioration in strength and serviceability. A durable material therefore, helps the environment by conserving resources and reducing wastes and the environmental impacts of repair and replacement. Attempt had been made in the past, to improve the durability characteristics of concrete by incorporating iron ore tailings in concrete. SiO<sub>2</sub> is the main oxide composition of the iron ore tailings, the oxide can constrain alkali silica reaction (ASR) in concrete. At 15 % replacement of sand by iron ore tailings, the ASR-expansion in concrete was reduced below 0.10 % [6]. A related study also revealed that self-compacting concrete (SCC) incorporating iron slag as partial replacement of fine aggregate, gives better strength and durability characteristics than the control SCC [24].

Different concretes require different degrees of durability depending on the exposure

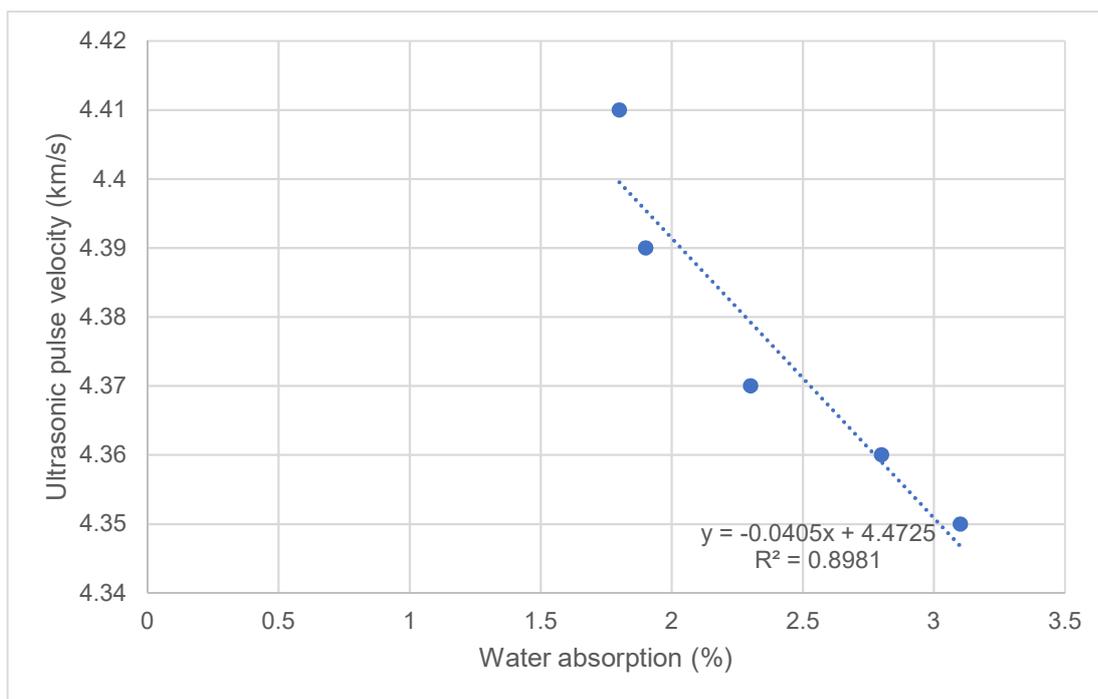


Fig. 12 - Relationship between water absorption and ultrasonic pulse velocity of IOTs concrete.

Table 5

Chemical requirements for natural pozzolans (ASTM, 2005)

	Mineral admixture class		
	N	F	C
Silicon dioxide (SiO <sub>2</sub> ) plus aluminum oxide (Al <sub>2</sub> O <sub>3</sub> ) plus iron oxide (Fe <sub>2</sub> O <sub>3</sub> ), min, %	70	70	50
Sulfur trioxide (SO <sub>3</sub> ), max, %	4.0	5.0	5.0
Moisture content, max, %	3.0	3.0	3.0
Loss on ignition, max, %	10.0	6.0	6.0
Available alkalis, equivalent of Na <sub>2</sub> O, max, %	1.5	1.5	1.5

environment and the properties desired. Moisture mainly enters a building through joints between concrete elements. Durability of concrete largely depends on the ease with which fluids can enter into, and move through, the concrete. This movement of the various fluids through concrete takes place not only by flow through the porous system but also by diffusion and sorption, so the main consideration is really with the penetrability of concrete. The penetrability of concrete is controlled by the bulk of the hardened cement paste which is the only continuous phase in concrete [1].

Due to the texture and particle size distribution of the iron ore tailings (IOTs) used in this study, the material was able to reduce the water permeable pore space in the IOTs concrete. From the test results, the correlation between the water absorption and ultrasonic pulse velocity of concrete samples was deduced as shown in Figure 12. It can be observed that there is strong inversely proportional relationship between water absorption and ultrasonic pulse velocity of iron ore tailings concrete. These results suggest that the small particle size of iron ore tailings was able to fill the pore space in concrete thereby producing more dense concrete, which gave higher values of ultrasonic pulse velocity and reduced water

absorption. The American Society for Testing and Materials (ASTM) requirements for Class N raw and calcined natural pozzolan, specify that, the total of three oxides (silica, alumina, and iron oxide) should not be less than 70 % [25] as indicated in Table 5. In comparison with ASTM chemical composition for natural pozzolan the total of the three oxides SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> in the iron ore tailings is 70.0 %, which satisfy the 70 % requirement for Class N raw and calcined natural pozzolan. The pozzolanic nature of the iron ore tailings used in this study, also contributed to better durability behavior of the IOTs concrete compared to the conventional concrete.

#### 4. Conclusion

The influence of iron ore tailings as fine aggregate to partially replace natural sand in normal strength concrete has been highlighted in this research. The contribution of iron ore tailings is beneficial to the sorptivity performance of normal strength concrete based on findings from the experimental works.

The greater the replacement level of iron ore tailings in concrete the lower the rate of water

absorbed by capillary suction implying that iron ore tailings can be used as fine aggregate to improve the water resistance of concrete. The material can be used to improve the absorption characteristics of the outer zone of concrete for the purpose of protecting the reinforcement.

The density and ultrasonic pulse velocity of concrete increased with more content of iron ore tailings thereby confirming the suitability of the tailings in reducing the volume of pore space in concrete. The material also contributes to increase, in compressive strength of normal strength concrete.

Flexural strength is very sensitive to defects such as micro-cracks and pores in the microstructure of concrete. There was significant linear correlation between flexural strength and sorptivity of the concrete. The iron ore tailings small particle size and being a well graded fine aggregate, is responsible for its efficiency in improving the packing density of the concrete.

Field emission scanning electron microscopy morphology of iron ore tailings and for the concrete samples containing iron ore tailings shows closely packed and dense microstructure compared to sand and reference concrete sample respectively.

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