Jurnal
Teknologi

Full Paper

EFFECT OF SULPHURIC ACID ON CONCRETE WITH IRON ORE TAILINGS

Article history
Received
24 May 2015
Received in revised form
20 November 2015
Accepted
22 December 2015

Ali Umara Shettima[a,c*], Mohd Warid Hussin[b], Yusof Ahmad[a], Mohammed Yau[c]

[a]Faculty of Civil Engineering, Department of Structures and Materials, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
[b]Construction Research Centre (UTM-CRC), Institute for Smart Infrastructure and Innovative Construction, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
[c]The federal Polytechnic Damaturu, Department of Civil Engineering Technology, Damaturu 620001, Yobe State, Nigeria

*Corresponding author
warid@utm.my

Abstract

Researches into the uses of waste materials are increasingly being explored to meet up society's needs and global protection for sustainable, safe and economic development. This paper assessed concrete with iron ore tailings (IOT) exposed to dilute sulphuric acid. Iron ore tailings are the materials left-over after separating the valuable fraction from the uneconomic fraction of an ore. To study the effect of sulphuric acid, concrete of 100 mm cube with a different mix ratios containing IOT were prepared and cured for 28 days in water. The cubes were later immersed into dilute sulphuric acid at a concentration of 5%. The compressive strength of concrete at 7, 28 and 90 days of water curing were determined. Mass loss and strength reduction due to sulphuric effect were evaluated at 7, 28 and 90 days respectively. XRD microstructure of concrete specimens was analysed. Test results indicated that the IOT could be used in concrete as sand replacement since the concrete with IOT has similar trend in compressive strength loss and mass loss to sulphuric acid attack compared to control specimen. The mineralogical crystal failure patterns due to the sulphuric acid in terms X-ray diffraction analysis are the same for control and IOT concrete.

Keywords: Concrete, Iron ore tailings, mass loss, compressive strength, sulphuric acid, X-ray diffraction

1.0 INTRODUCTION

Concrete is one of the most widely used construction materials and it offers numerous advantages; among which is its ability to be moulded to various shape, ease of handling and its durability. Over the several past decades, the demand for concrete has increased rapidly due to growth in infrastructural development. Hence, the demands for raw materials were raised. River sand is one of the main materials in concrete production as it is used as fine aggregate. The heavy demand for river sand for concrete production has resulted in the over-exploitation of river bed, which leads to a range of problems including increased river bed depth, water table lowering and intrusion of salinity into ground water [1]. As such, finding and supplying alternatives for river sand has become imperative. This would generate sustainable concrete and greener environment [2]. One of the alternatives for the replacement of river sand is the utilization of industrial wastes. The use of industrial waste in concrete making is gaining recognition in order to reduce environmental pollution and cost of river sand that was hiked due to the restriction in its extraction for sustainability [3].

It is widely reported that concrete is prone to sulphuric acid attack which causes both dissolving and swelling [4]. Sulphuric acid are usually formed due to the reaction of sulphur in the ground and ground water. It is also produced from sewage facilities and sulphur dioxide present in the atmosphere [5]. Chemically, sulphuric acid is aggressive and reacts easily with lime to form gypsum. This will lead to swelling, expansion and cracks as a result of internal pressure [3].

The recent increase in the production of iron ore to meet up to the demand of steel industries has generated massive iron ore tailings. These tailings have serious environmental impact apart from occupying large area of landfill disposal [6]. Research works on iron ore tailings for concrete infrastructure are on the increase. They were used for cements replacement as well as fine aggregate in concrete or mortar production. However, majority of the research work focused on mechanical properties. There is limited research data about their performance on exposure to sulphuric acid.

2.0 MATERIALS

British standard CEM I ordinary Portland cement [7] obtained from Tasek cement manufacturing company of Malaysia was used. Natural river sand and crushed granites were used as fine and coarse aggregates respectively. Iron ore tailings with specific gravity of 2.6, relative density 1.27 g/cm^3, fineness modulus of 1.05 and water absorption rate of 7.0% were obtained from one iron ore mill in Johor, a southern State in Malaysia Peninsula. The chemical composition of natural sand and iron ore tailings were presented in Table 1.

A superplasticizer of Polycarboxylic ether based (Rheobuild) 1100 complying with ASTM C494 [8] was used.

3.0 MIX PROPORTION

The mix proportions were designed according to BS EN 206 [9] standard, aggressive exposure class XA1 for grade C37 concrete as shown in Table 2.

Five mix proportions were prepared. The first was control mix which is a conventional concrete designated as NC (i.e. without iron ore tailings), and the other four mixes contained iron ore tailings. The fine aggregate (sand) was replaced with iron ore tailings by mass. The amount of replacements were25%, 50%, 75% and 100% and designated as M25, M50, M75 and M100 respectively. For each concrete mixture, test specimen of 100mm cubes were cast and cured for 28 days under water at $27^0 \pm 2^0$ C until the test age

Table 1 Chemical composition of river sand and iron ore tailings

Chemical composition (%)	SiO	Al_2O_3	Fe_2O_3	CaO	MgO	MnO	K_2O	CUO	ZnO	PbO	SO_3	LOI
Sand	98	0.4	0.1	0.9	0.2	0.02	1.4	-	-	-	-	0.1
IOT	56	10	8.3	4.3	-	1.7	1.5	0.2	0.1	0.4	-	3.3

Table 2 Mix proportion of concrete mixes incorporating iron ore tailings

		Proportion of IOT's			
Description	(NC)	M25	M50	M75	M100
OPC (kg/m^3)	380	380	380	380	380
Coarse aggregate (kg/m^3)	900	900	900	900	900
Natural sand (kg/m^3)	830	622	415	208	-
Iron ore tailings (kg/m^3)	-	208	415	622	830
Water (kg/m^3)	190	190	190	190	190
Superplastiizer (%)	1.0	1.0	1.0	1.0	1.0
Slump (mm)	130	110	100	90	80

4.0 EXPERIMENTAL PROGRAM

Concrete cubes of 100 mm were prepared and cured in water for 28 days before putting them into 5% dilute sulphuric acid (H_2SO_4) solution. Prior to immersion, the cube specimen were wiped to surface dry and weighed to the nearest gram using electronic weighing balance and this was noted as the reference weight. The pH of the solution was regularly monitored and adjusted to keep constant by replacing the consumed solution with a fresh solution with the aid of pH meter. The assessment of normal concrete and those of the IOT specimens in acidic environment was based on the mass loss, compressive strength, strength loss factor and X-ray diffraction analysis. All these were determined at 7, 28 and 90 days of immersion. The crystal spectrums of concretes at 90 days in sulphuric acid were examined using X-ray diffraction techniques. XRD diffractometer D5000 machine of Siemen Company was used for the tests. X-ray test was conducted at a source of Cu Ka with 40 KV of X-ray acceleration voltage and 30 mA electric current. The scanning rate is 0.05^O/sec with a scanning interval of 2Θ angle from 5 ^{O}C to 65 ^{O}C.

5.0 RESULTS AND DISCUSSION

5.1 Compressive Strength

The compressive strength tests of conventional concrete and iron ore tailings concrete at 7, 28 and 90 days cured in water are shown in Figure 1. The results show that the compressive strength of IOT concrete specimens were higher compared to that of the control specimens. Concrete specimen M50 displayed the highest strength throughout the curing period. However, with the increase in percentage of IOT, the compressive strength equally decreased. Zhang, *et al*. [10] equally observed similar trend as IOT addition enhanced strength increments in relation to NC, but decreased with increased in IOT percentage. The observed higher compressive strength of IOT concretes could be partly attributed to the filler effect of fine particles of the tailings which has contributed to strength enhancement witnessed. It is equally suspected that the presence of iron in higher concentrations has positive impact on the strength attainment [11].

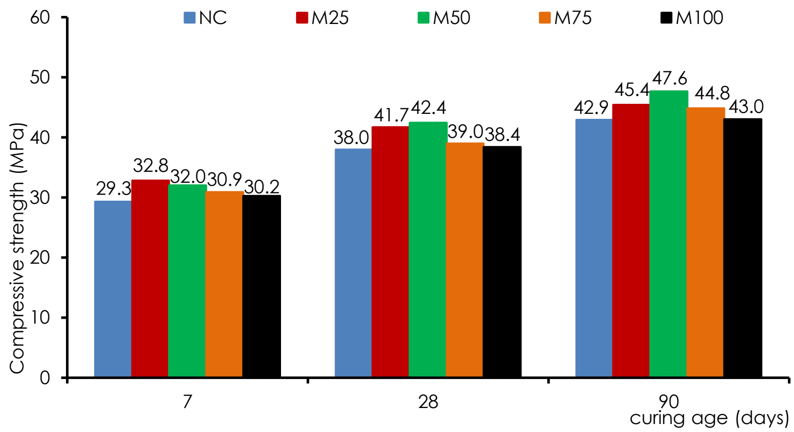

Figure 1 Compressive strength of concrete specimens cured in water

5.2 Mass Loss

The effect of sulphuric acid on the NC and IOT concrete samples on the mass are presented in Figure 2. It is observed that the entire specimens exhibit the same trend (decrease in mass) throughout the period of 7, 28 and 90 days of immersion. This loss is brought about by the action of the acid which reacts with calcium hydroxide to damage the cement gel binder of concrete [12] and formed white soft and soluble gypsum on the surface. It is also observed that throughout period of exposure, the weight of the IOT concretes decreased more than the conventional concrete (control). This could be attributed to the fineness of the IOT and direct attack on the aluminosilicate framework by breaking the bonds. The alumina bonds are more prone to breakage due to their weakness and this leads to less aluminate on the composition of the IOT concrete more than it does to the normal concrete. The stronger silica bonds of river sand in the normal concrete tend to resist the attack by acid more than alumina bond of IOT concrete.

5.3 Compressive Strength Loss

Figure 3 presents the compressive strength loss of specimens immersed in sulphuric acid solution. The strength loss associated with the entire samples is the consequences of sulphuric acid. It could be seen that NC and M_{100} loses 57% and 47% compressive strength respectively at 90 days of immersion in acid compared with 90 days compressive strength in water curing. This could be attributed to the finer particles which occupied the pore space and form dense concrete. The destructive action of acid is as a result of the reaction between calcium and aluminate in the composition forming the less soluble reaction product ($3CaO.Al_2O_3.2H_2O$). This very expansive compound causes internal pressure in concrete, which results in the formation of cracks and surface softening and results in the loss of mechanical strength of concrete.

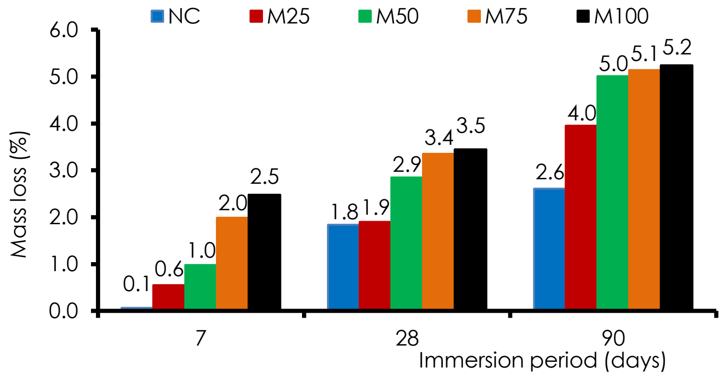

Figure 2 Mass loss of concrete specimens immersed in sulphuric acid solution

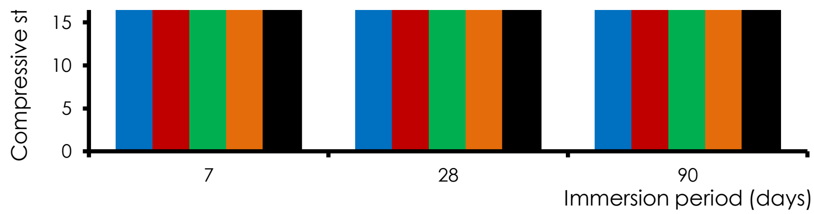

Figure 3 strength loss of concretes specimen immersed in sulphuric acid solution

5.4 Strength Loss Factor

Strength loss factor is a quantitative way of expressing the performance of IOT concrete in the state of qualitative means. The deterioration of concrete specimens was investigated by measuring the strength loss factor (SLF) expressed in percentage and was calculated using Equation 1.

$$SLF = \frac{Fcw - Fca}{F_{Ca}} x100\% \qquad (1)$$

Where:

SLF = strength loss factor

F_{CW} = average compressive strength of companion specimen cured in water.

F_{Ca} = average compressive strength of the specimen after immersion in acid solution

The reduction in the compressive strength of normal concrete specimens and the IOT due to acid attack was expressed in the form of SLF at the period of 7, 28 and 90 days of immersion in acid as presented in Figure 4. The normal concrete specimens are severely affected by the acid during the 90 days of immersion and hence the SLF value was higher, compared to SLF values observed in the IOT concrete specimen under the same condition. It could be seen that the addition of IOT in concrete specimens reduces the micro pores and thus form a dense concrete mass.

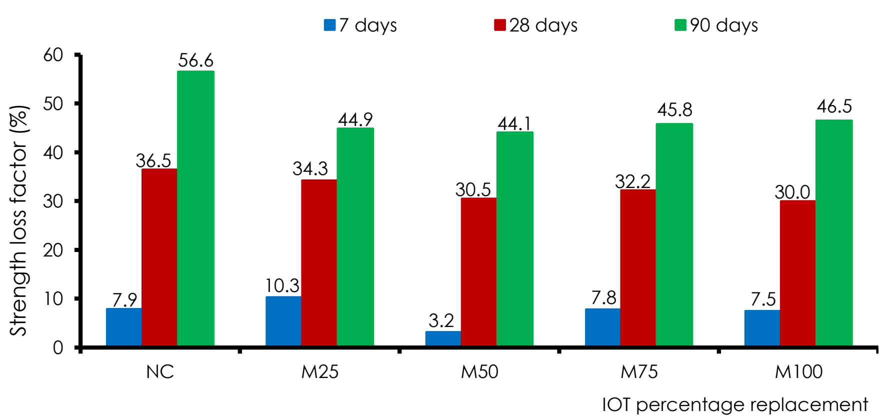

Figure 4 Strength loss factors of normal concrete and IOT concrete

5.5 XRD Analysis

Figure 5 exhibits the XRD patterns of concrete specimens at 90 days in sulphuric acid exposure. The results show that there is no significant difference between the behaviour of IOT and NC concrete exposed to sulphuric acid. The dominant crystals that were evident due to the reaction of acid are gypsum, ettringite and calcite. The concrete specimen of M_{25} does not have enttringite. Traces of portlandite are noticed in normal concrete (control), while melanterite [$Fe^{2+}SO^{4}$ 4($H^{2}O$)] is identified in the concrete with iron ore tailings. Melanterite is a hydrated iron sulphate formed after the decomposition of IOT due to the action of sulphuric acid.

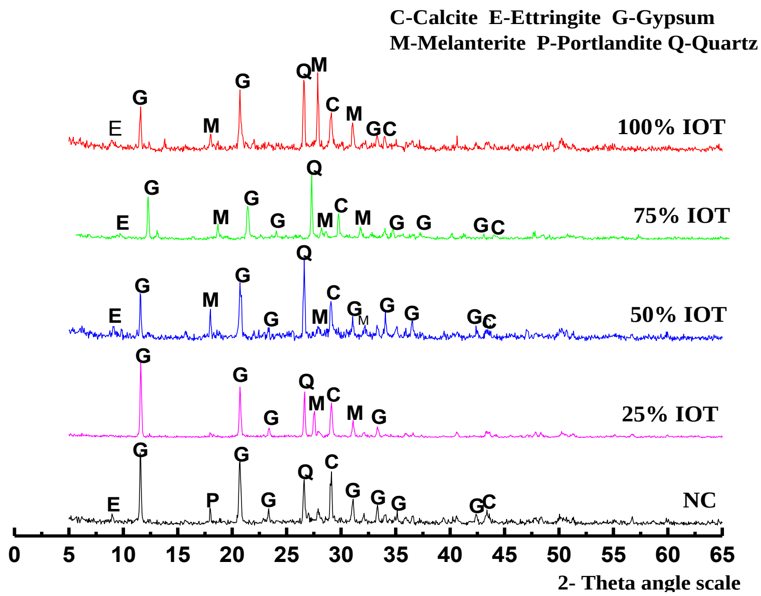

Figure 5 XRD patterns of concrete specimens at 90 days sulphuric acid exposure

6.0 CONCLUSION

Based on the test results the following conclusions can be summarised:

1. Iron ore tailings concretes have consistently higher compressive strength than reference concrete. They all attained the designed strength at 28 days curing.
2. The weight loss of IOT concrete specimens are considerably higher compared to reference conventional concrete at 90 days of acid exposure.
3. The residual compressive strength of concrete with iron ore tailings after exposure to acid is higher than the reference concrete. This indicates that concrete with iron ore tailings can withstand acid to some degree of exposure and can be used for fine aggregate for sustainability and environmental protection.
4. XRD examination reveals the formation of gypsum, ettringite and melanterite due to the reaction of acid and this has generated the loss of mechanical strength and mass loss.

Acknowledgement

The authors are grateful to Ministry of Education (MOE) and Research Management Centre (RMC), Universiti Tecknologi Malaysia (UTM) for providing the Research University Grant (RUG), Vot. No: Q. J13000. 2509. 06H56, Q.J130000.2422.02G84 and R.J130000. 7722.4J127.

References

[1] Ganesh Prabhu, G., J. H. Hyun and Y. Y. Kim. 2014. Effects Of Foundry Sand As A Fine Aggregate In Concrete Production. *Construction and Building Materials.* 70(514-521.

[2] Senthamarai, R. M., P. D. Manoharan and D. Gobinath. 2011. Concrete Made From Ceramic Industry Waste: Durability Properties. *Construction and Building Materials.* 25(5): 2413-2419.

[3] Aydın, S., H. Yazıcı, H. Yiğiter and B. Baradan. 2007. Sulfuric Acid Resistance Of High-Volume Fly Ash Concrete. *Building and Environment.* 42(2): 717-721.

[4] Raju, P. S. N. and P. Dayaratnami. 1984. Durability of Concrete Exposed to Dilute Sulphuric Acid. *Building and Environment.* 19(2).

[5] Girardi, F. and R. D. Maggio. 2011. Resistance Of Concrete Mixtures To Cyclic Sulfuric Acid Exposure And Mixed Sulfates: Effect Of The Type Of Aggregate. *Cement and Concrete Composites.* 33(2): 276-285.

[6] Li, C., H. Sun, J. Bai and L. Li. 2010. Innovative Methodology For Comprehensive Utilization Of Iron Ore Tailings: Part 1. The Recovery Of Iron From Iron Ore Tailings Using Magnetic Separation After Magnetizing Roasting. *Journal Of Hazardous Materials.* 174(1-3): 71-77.

[7] EN197-1. 2000. European Standard Specification For Cement: Composition, Pecifications And Conformity Criteria For Common Cements. BSI Standards Publication.

[8] ASTM C494. 2013. American Standard Testing of Materials - Standard Specification for Chemical Admixtures for Concrete. American Society Of Testing And Materials Publication.

[9] EN206-1. 2013. European Standard For Concrete: Specification, Performance, Production And Conformity. BSI Standards Publication.

[10] Zhang, G. D., X. Z. Zhang, Z. H. Zhou and X. Cheng. 2013. Preparation and Properties of Concrete Containing Iron Tailings/Manufactured Sand as Fine Aggregate. *Advanced Materials Research*. 838-841: 152-155.

[11] Yellishetty, M., V. Karpe, E. H. Reddy, K. N. Subhash and P. G. Ranjith. 2008. Reuse Of Iron Ore Mineral Wastes In Civil Engineering Constructions: A Case Study. *Resources, Conservation and Recycling.* 52(11): 1283-1289.

[12] Bakharev, T., J. G. Sanjayan and Y. B. Cheng. 2003. Resistance Of Alkali-Activated Slag Concrete To Acid Attack. *Cement and Concrete Research.* 33(10): 1607-1611.