Evaluation of Mechanical Properties of Unsaturated Polyester Resin Composite Tiles of Granite Quarry Dust Filler

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Abstract - The effects of granite quarry dust content and particle size on the mechanical properties of unsaturated polyester resin composite tiles produced by a simple casting process was evaluated. The quarry dust content employed was within 0 to 80 wt. % while the particle sizes of the quarry dust used in the formulations were 53, and 150 µm respectively. The mechanical properties of the composite tile samples were characterized by standard methods. Result showed a general decrease in tensile strength of the composite tile samples with granite quarry dust content. The composite tile sample formulated with quarry dust of 53 µm particle size exhibited the highest tensile strength of 46.73 MPa at 20 wt. % quarry dust content. The composite tile samples exhibited maximum elongation at break (13. 0%) at 20% quarry dust content. Generally, there was a general decrease of elongation at break with increase in granite quarry dust content. The tensile modulus of the polyester resin (420 MPa) without quarry dust was greater than those containing quarry dust at all quarry dust content and particle size investigated. The hardness of the composite tile samples increased with increasing content of granite quarry dust and was higher than the hardness of the sample without quarry dust (7.5 HV). The samples containing granite quarry dust (150 µm) generally exhibited higher hardness than those of granite quarry dust of particle size, 50 µm, and at 60 wt. % granite quarry dust content, the samples had nearly the same hardness, 15.60 and 15.70 HV respectively. The present study has highlighted the good hardness property, impact strength, and tensile strength (46.73 MPa obtained with dust of particle size, 53 µm) of the formulated tile samples. These results should justify the use of quarry dust in the preparation of unsaturated polyester resin composite tiles, at least, in formulations where these properties are of paramount importance.

Index Terms - Composite, elongation at break, granite quarry dust, impact strength, tensile strength, tiles, unsaturated polyester resin.

1. Introduction

A tile is a manufactured piece of hard material such as ceramic, stone, metal or even, glass. Tiles are used in both interior and exterior decoration to cover roofs, floors, walls in houses, roofing, gutters, and public buildings. Tiles are also used in artificial applications such as designing an art work, and applied in space shuttle to protect the shuttle and its occupants from high temperature experienced when the shuttle re-enters the earth atmosphere during landing. The production of tiles dated back to ancient times, and people (including Egyptians, Babylonians, Assyrians, etc.).

Europe, Latin America and the Far East are the largest producers of tiles, with Italy as the leader at 16.6 million ft^2/day as at 1989. Following Italy (at 24.6 % of the world market) are Spain (12.6 %), Brazil and Germany (both at 11.20 %) and the United States (4.50 %). The total market for floor and wall tile in 1990 according to one estimate was 2.46 billion.

Tiles can generally be classified into three basic types namely: ceramic tile, porcelain tile, and glass tile [1]. All ceramic tiles including porcelain are made of clay, some additives (such as feldspar and quartz sand), and water. Porcelain tile is generally made by the dust pressed method using porcelain clays resulting in tile that is dense, impervious, fine grained and smooth with a sharply formed face[2]. Porcelain tiles usually absorb less water (< 0.5 %) than non - porcelain tiles, and this makes them frost resistant or frost - proof. These tiles are available in matte, unglazed or a polished finish. Glass tiles, on the other hand, contain pieces of glass formed into consistent shapes.

Tiles can be glazed or unglazed. Glazed tiles are coated with a thin layer of glass while unglazed tiles do not have coated surfaces and body but can be specially treated to alter the surface appearance, thus, producing tiles that can be polished, semi-polished, lapped and satin-finished [3].

There are several methods of producing tiles, and these include dry pressing, extrusion, punching, ram pressing and pressure glazing [4]. The raw materials used to form tiles consist of clay minerals obtained from the earth crust, polyester resins, natural minerals such as feldspar, and other chemical additives.

Different natural materials have been investigated as fillers for the production of composite tiles. Thus, Martinez-Barrera[5] studied the effect of marble particle size on the compressive properties of polyester tiles, and found that tiles produced using marble of particle size 1.40 mm had the higher compressive strength (90 MPa). Maksimor et al.[6] produced polyester resin tiles using granite (58 wt. %), limestone (10.4 wt. %) and sand (21.8 wt. %). The resulting tile had tensile strength in excess of 80 MPa.

Icduygu et al.[7] fabricated composite tiles using poly(ethylene terephthalate), micro-marble particles reinforced with glass fibre mats, recycled or commercial resin and reported that tiles prepared using marble particles generally gave higher flexural strength and stiffness values irrespective of whether synthesized or commercial resin was used or not. On the other-hand, the flexural strength and stiffness of the tiles prepared using commercial polyester resin was observed to be 1.8, and 2.5 times higher than those prepared using synthesized resin. Tawfik and Eskander [8] produced polyester resin tiles using marble clusters of grain sizes, 1 to 5 mm, styrene, and a co - monomer, and the resin tile produced had a compressive strength of 120 MPa at styrene optimum composition of 60 %.

Bozadgier and Popova[9] prepared perlite based ceramic tiles using refractory clay, quartz, sand and sodium feldspar and found that the substitution of sodium feldspar by perlite in the production of ceramic tiles increased not only the mechanical strength and shrinkage of the tile samples but also, decreased the water absorption capacity of the samples. It has been reported that ceramic tile bodies containing 15 – 25 wt. % perlite, 55 –

60 wt. % refractory clay, and the weight balance of glass cullets after firing at 950 ^{0}C had 44.3 – 46.1 MPa bending strength, 7.5 – 8.1 % water absorption, and 4.2 – 5.4 % shrinkage [10].

Achukwu *et al*.[11] fabricated waste glass/ unsaturated polyester resin composites at glass contents 5, 10, 20, 30, 40, 50, and 60 wt. % using the hand lay – up method. The glass was of particle sizes 75, 150, and 212 µm. The tensile and impact strength of the composites was observed to increase with glass content. The 75, and 150 µm waste glass fabricated composites exhibited the highest tensile strength at 20, and 5 wt. % filler content. Onuegbu and Amadi[12] investigated the properties of unsaturated polyester resin concretes made from sand contaminated with crude oil and reported that crude oil contamination reduced the compressive strength of polymer concretes. This was attributed to the effect of contamination on the 3 – D crosslinking of the polymer or it's curing. Ibeneme *et al*.[13] utilized huracrepitan pod fibre to fabricate unsaturated polyester resin tile samples having improved mechanical properties. The impact strength, elongation at break, and water absorption of the tile samples were observed to increase with increasing filler content.

Although, polyester resin tiles have wide range of applications, they are characterized by brittle failure and limited toughness [5], [13], [14]. These drawbacks have been addressed by studying the effect of filler particle size, and chemical composition of the polyester resin tiles. Furthermore, the use of more than one reinforcing fillers have been suggested in addressing the observed shortcomings of polyester resin tiles [7].

There is presently scarcity of fine aggregates in many locations for building and construction purposes and this has worsened the shortage for the increasing demand for concrete and tiling products. Many sand mining sites have posed challenges such as the deepening of river valleys which sometimes, lead to washouts of pile foundations supporting bridges. It is a welcome idea to partially replace marble dust and sand with other materials such as granite quarry dust in the production of concrete products. Fortunately, the establishment of rock processing sites produces great volume of granite quarry dust which can be utilized for industrial products.

In the present report, unsaturated polyester resin composite tiles were produced using granite quarry dust collected from Conrok Quarry, Afikpo, Nigeria. Granite quarry dust is a by – product of rock quarrying processing with a large proportion of its particle size being far less than 200 µm in size. The granite quarry dust was sieved to 53, and 150 µm particle sizes, and used within filler contents, 0 to 80.0 wt. % in formulating the unsaturated polyester resin composite tiles. Unsaturated polyester resin was used as a matrix in this study because of its wide range of mechanical properties coupled with its low cost, molecular weight, and corrosion resistant property. Generally, unsaturated polyester resins are used in fabricating industrial products that have large applications.

2. Materials and Methods

2.1 Materials

The following materials were used in this study.

Granite quarry dust was collected from Conrok Quary, Afikpo, Nigeria and used as a filler. The medium oil soya bean alkyd resin used as a matrix was obtained from a chemical store at Onitsha, Nigeria and has an acid number 6.13 mgKOH/g resin. Methyl ethyl ketone peroxide (MEK) was purchased from Campal Scientific and Technological Co. Ltd, Onitsha, Nigeria and used as an initiator while cobalt naphthanate obtained from Tonyker Nigeria Ltd, Onitsha, Nigeria was used as an accelerator. Silicon oil was used as a mould release agent.

2.2 Compositional Analysis of Granite Quarry Dust

The chemical composition of granite quarry dust was determined using ASTM D 5381 – 93 method. An energy dispersive X – ray Spectroscopy (EDXRS) (Phenom Prox.) was used in the analysis.

2.3 Preparation of Quarry Granite Dust

The quarry granite dust collected from a quarry processing plant was sun dried and impurities were carefully removed from the aggregates before crushing to fine powder to get the quarry dust. The dust was calcined in a hot oven at 900^{o}C for 3 hr after which it was removed from the oven, cooled, and sieved to 53, and 150 µm mesh sizes. The sieved dust was stored in a tight lid container for subsequent use.

2.4 Preparation of Unsaturated Polyester Resin Composite Tiles

The granite quarry dust of particles sizes 53, and 150 µm were used in preparing unsaturated polyester resin composite tiles. Six samples of unsaturated polyester resin composite tiles having a composition of unsaturated polyester resin to quarry dust ratios of 98:0, 88:10, 78:20, 68:30,58:40, 48:50, and 38:60 were prepared. An unsaturated polyester resin containing no granite quarry dust served as a control.

In the blank preparation containing without quarry dust, 1g of cobalt naphthanate was added to 98 g of the unsaturated polyester resin and the mixture was stirred for 2 min after which 1 g of methyl ethyl ketone was added. The mixture stirred was stirred for 1 min before pouring into a mould (17 x 17 x 4 cm) prepared by adding silicon oil on its inner surfaces. The sample was placed in a safe place and allowed to cure for three days after which it was demoulded and stored in a tight lid container for further use.

In other preparations, unsaturated polyester resin composite tile samples containing granite quarry dust filler were prepared as above using the stated resin to sand ratios for the two filler particle sizes. The amount of methyl ketone peroxide used was within acceptable limit since it has been reported that amounts in excess of 3% of the quantity of resin used results in degradation of mechanical properties and glass transition temperature [17].

2.5 Testings on Prepared Tile Samples

The following tests were carried out on the prepared tile samples using standard methods: impact strength (ASTM D 6110), hardness (ASTM E 384). The tensile strength, tensile modulus and elongation at break of the tile samples were obtained from tensile testings using ASTM D 638 method. A Universal testing machine (Instron Series, 3369) with an operation range, 1 – 500 mm/min was used to carry out the tests. From the testings, the following parameters were obtained:

Tensile modulus was calculated from the ratio of the stress to strain where,

$$\text{Stress} = \text{load/area} \qquad (2.1)$$

$$\text{Strain} = \text{elongation/original length} \qquad (2.2)$$

The elongation at break of thee samples was calculated as,

$$\% \text{ Elongation} = [\,(l_1 - l_0)\,] / 100 \qquad (2.3)$$

where:

l_0 = original length of specimen (cm).

l_1 = final length of specimen (cm).

3. Results and Discussion

3.1 Chemical Composition of Granite Quarry Dust

The composition of granite quarry dust determined using energy dispersive X – ray spectrometer showed the abundance of silicon dioxide (59.62 %). Silicon dioxide ($Si0_2$) which is chemically inert, and non – toxic is the most common mineral that is resistant to weathering. Other oxides present in varying proportions are aluminum oxide (13.92 %), potassium oxide (7.98 %), calcium oxide (5.14 %), iron (iii) oxide (3.00 %), copper oxide (2.55 %), silver oxide (2.14 %), sodium oxide (1.16 wt. %), phosphorus oxide (1.86 wt. %), magnesium oxide (1.21 wt. %), and sulphur oxide (1.42 wt. %)

3.2 Determinations on the Prepared Tile Samples

3.2.1 Tensile Strength

Data obtained on the effect of unsaturated polyester resin and quarry dust contents on the tensile strength of the prepared composite tile samples are illustrated in Fig. 1. The figure shows that the tensile strength of the tile sample containing no granite quarry dust was higher than those of the samples containing granite quarry dust except for the tile sample containing 20 wt. % granite quarry dust of 53 µm particle size which exhibited the maximum tensile strength (46.73 MPa). Tile samples containing quarry dust of 53, and 150 µm particle sizes had the same tensile strength at 40, and 60 wt. % quarry dust content, and which was the least tensile strength recorded for all the formulations.

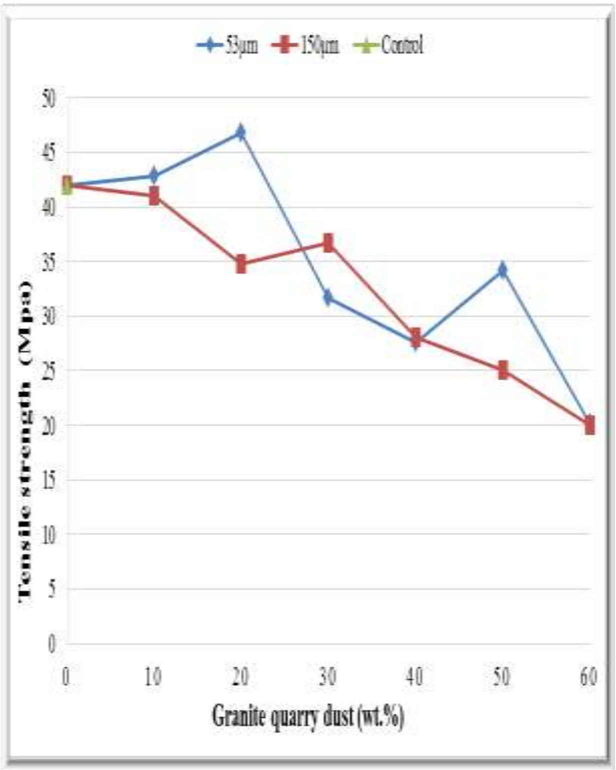

Fig. 1**:** Effect of granite quarry dust content and particle size on the tensile strength of formulated tile samples.

3.2.2 Elongation at Break

Data obtained on the elongation at break of the unsaturated polyester resin composite tiles are illustrated graphically in Fig. 2. The figure shows a general decrease of the tile elongation at break with filler content. The elongation at break of the cured polyester resin sample was higher than those of filled tile samples. The decrease of elongation at break with filler content is attributed to the stiffening and hardening of the tile samples due to the incorporation of quarry dust. This results to the reduction of resilience and toughness of the tile samples, thereby, lowering the elongation at break. The decrease of elongation at break with filler content indicates that the added quarry dust was not able to support the stress transferred from the dust particles to the polymer matrix. This, in effect, led to the

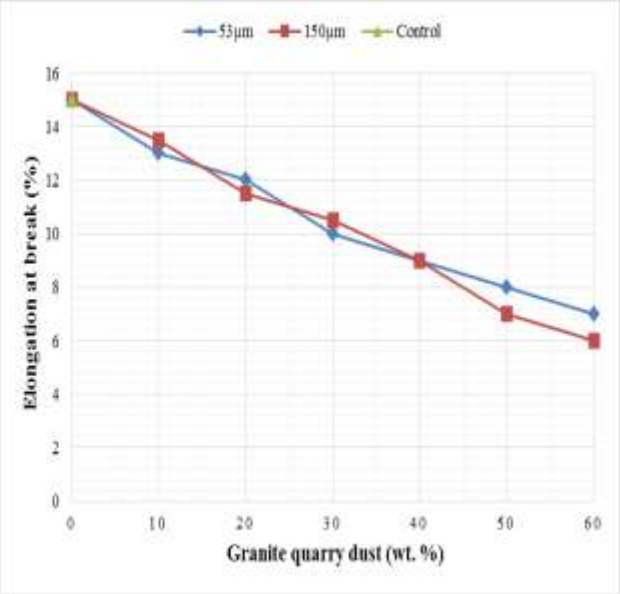

Fig. 2: Effect of granite quarry granite dust content and particle on the elongation at break of formulated tile samples.

decrease of elongation at break of the samples with increasing stiffness of the dust particles. Similar reduction of elongation at break of unsaturated polyester resin composite tile samples with filler content was reported by Ibeneme *et al.*[13] who reported that the elongation at break of unsaturated polyester resin/ huracrepitan pod fibre decreased with increasing filler content. The elongation at break of the tile samples formulated with granite dust of 53, and 150 µm particle sizes were quite similar, though, at filler contents above 50 wt.%, the tile samples containing granite dust of 53 µm particle size had higher elongation at break than those of 150 µm particle size.

3.2.3 Modulus

Fig. 3 illustrates the effect of granite quarry dust content and particle size on the modulus of the prepared tile samples. There was a general decrease of modulus with granite dust content. The formulations did not exhibit any definite order of variation will filler content. The 150 µm quarry dust formulated tile sample had the highest modulus (372.91 MPa) at 10.0wt. % filler content while the 53 µm granite dust formulated tile sample had the least modulus (223.78 MPa) at 80.0 wt.% filler content. The modulus of 150 µm granite dust formulated tile samples was generally greater than those of 53 µm granite dust formulated tiles samples. The modulus of tile sample containing no granite quarry dust was higher than the formulations containing granite quarry dust. The observed decrease of modulus of formulated tile samples with increasing granite quarry dust content may be due to insufficient adhesion between the granite quarry dust particles and the unsaturated polyester resin matrix. Adhesion is an important property that promotes good mechanical property of composites.

Fig. 3: Effect of granite quarry dust content and particle size on the modulus of formulated tile samples.

3.2.4 Hardness

The hardness of the tile samples was measured with Vickers hardness tester (MVI – PC, 07/2010 – 1329). Fig. 4 shows the plot of hardness of unsaturated polyester composite tile samples as a function of granite dust and unsaturated polyester resin contents. There was a general increase of sample hardness with increase in granite quarry dust content. The hardness of sample containing no granite quarry dust was generally lower than those of

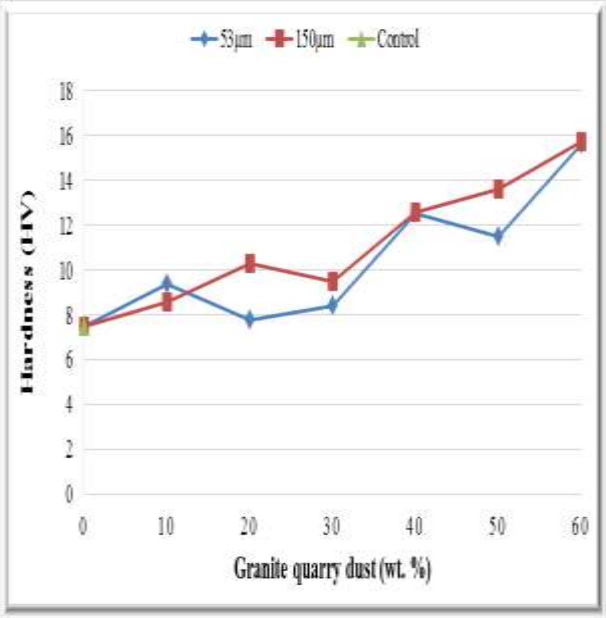

Fig. 4: Effect of granite quarry dust content and particle size on the hardness of formulated tile samples.

samples containing granite quarry dust. The hardness of tile sample containing granite quarry dust (150 µm) was generally higher than those of dust particle of size, 53 µm. The general increase of the modulus of tile samples is attributed to the reinforcing effect of the added granite quarry dust particles.

The result of this study is in agreement with the findings of Ibeneme *et al*.[13] who reported a general increase in hardness of unsaturated tile samples with filler contents. On the other hand, Awad *et al* .(2019) reported an increase of composite hardness with decreasing filler content. Hardness is a property that depends on strength, toughness, ductility, elasticity, and stiffness of materials.

3.2.5 Impact Strength

The impact strength of the tile samples determined using Charpy impact tester (CAT. NR, Nr. 412) as a function of granite quarry dust content and particle size is shown in Fig. 4.5. Granite quarry dust (53 µm) formulated tile samples exhibited a general increase of impact strength with granite dust content and has the highest impact strength (2.2 J) at 60 wt. % granite quarry dust content. The granite quarry dust (150 µm) formulated tile sample exhibited high impact strength (1.7 J) at 10 wt. % after which the impact strength decreased with filler content but was generally higher than the impact strength of the tile sample without granite quarry dust (0.2 J).

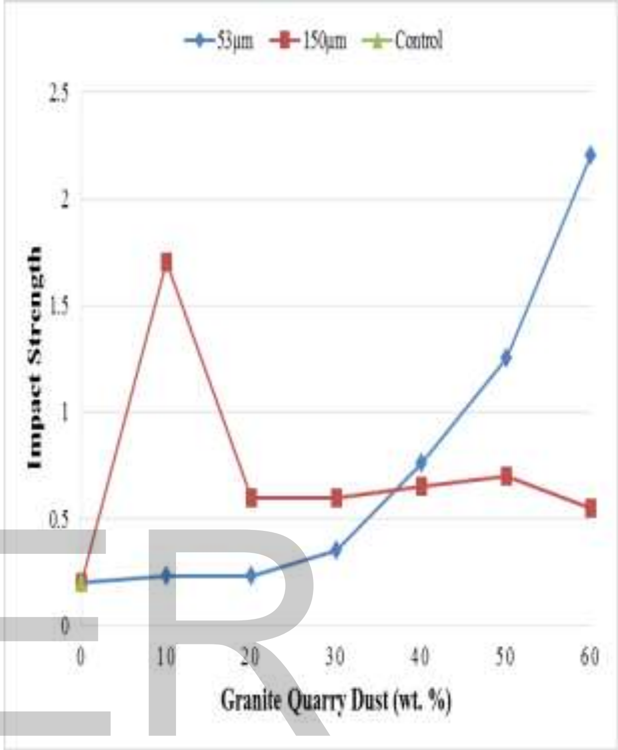

Fig. 5. Effect of granite quarry dust content and particle size on the impact strength of formulated tile samples.

4. Conclusion

Granite quarry dust obtained from Umuahia, Nigeria has been used to prepare unsaturated polyester resin composite tiles with the following properties.

The unsaturated polyester resin composite tile sample containing granite quarry dust of particle size 53 µm exhibited the highest tensile strength (46.73 MPa) when compared to the other formulations. However, the formulation without granite quarry dust generally had higher tensile strength than the formulations containing granite quarry dust except for the tile sample containing quarry dust of particle size, 153 µm at 10, and 20 wt. % granite quarry dust contents.

The formulated tile samples exhibited lower elongation at break compared to the formulation without granite quarry dust. At filler contents above 50 wt.%, the formulated tile samples containing granite quarry dust of particle size, 50 µm exhibited higher elongation at break than those of formulations containing granite quarry dust of particle size, 150 µm.

The modulus of formulated tile samples decreased with increasing granite quarry dust content, and was generally lower than that of the formulation without granite quarry dust (420.0 MPa). The highest modulus of 372. 90 MPa was obtained with granite quarry dust of 150 µm particle size at 10. 0 wt. % filler content.

The impact strength of granite quarry dust(53 µm) formulated unsaturated polyester resin tiles increased with increasing filler content, and had the highest strength (2.2 J) at 60 wt. % filler content while the impact strength of granite quarry dust (150 µm) decreased with filler content with a maximum strength (1.7 J) at 10 wt. % filler content.

The hardness of the formulated tile samples were found to increase with increasing granite dust content and the hardness of tile samples containing granite quarry dust (150 µm) were generally greater than those containing granite dust of particle size, 50 µm.

The unsaturated polyester resin composite tiles prepared using granite quarry dust in this study showed that the quarry dust content and particle size greatly influenced the tile properties. The samples containing granite quarry dust exhibited high hardness, high tensile strength (42.78, and 46.78 MPa) obtained with quarry dust of 53 µm particle size, and good impact strength, also, obtained with granite quarry dust (53 µm). These results should justify the use of granite quarry dust in the composite industry where these enhanced properties are the ultimate requirements.

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