IJNRD.ORG ISSN : 2456-4184
INTERNATIONAL JOURNAL OF NOVEL RESEARCH AND DEVELOPMENT (IJNRD) | IJNRD.ORG
An International Open Access, Peer-reviewed, Refereed Journal

Investigation on the Effect of Iron Oxide (Fe_2O_3) Pigment on the Production of Coloured Cement for Concrete Works

J.S. Okoh, Dr. P.E. Dim
Department of Chemical Engineering, School of Infrastructure and Process Engineering Technology, Federal University of Technology, Minna Niger State, Nigeria.

Abstract: Coloured concrete has gained significant popularity in Engineering and decorative applications due to its aesthetic appeal. Iron oxide (Fe_2O_3) pigments are widely used to impart colour to cement-based materials. This research work investigates the effect of Fe_2O_3 pigment on the production of coloured cement for concrete works. It explores the influence of Fe_2O_3 pigment on concrete properties, such as compressive strength, durability, absorption rate and colour stability. The research involves a series of experimental analyses, including Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), and Scanning Electron Microscopy (SEM). Results indicate that a 3:50 ratio of Fe_2O_3 pigment to cement achieves the desired color change without significantly altering the chemical composition or crystalline structure of the cement. The effects of these pigments on concrete properties were studied on Portland cement, the strengths of the concrete stones were studied by compressive strength test with an average strength of 5.885 N/mm² and 5.878 N/mm² for 3-day cured samples, and 6.558 N/mm² and 6.611 N/mm² for 28-day cured samples, respectively. The durability studied through water absorption with an average value of 6.96%, as well as loss of igniton, blaine fineness, initial and final setting time of cement were determined, with achieving a 100% colour retention which is imbedded in the concrete material. It is found from this work that coloured pigments does not affect concrete works when applied but enhance compressive strength, inbuilt colour that is not just on the peripheral but imbedded inside the concrete.

Keywords: Cement, Pigments, Iron oxide (Fe_2O_3), Colour retention

1 Introduction

Cement is the most widely used and adaptable material in the construction industry for its properties and cost (Heerah *et al.,* 2020). Despite its numerous qualities, the production of cement contributes largely to the release of carbon dioxide (CO_2) in the atmosphere and, its structures have a dull grey appearance (Yun *et al.,* 2019).

Concrete appearance is affected by many factors, including the ingredients in the mix, mix design, handling and placing procedures, forming and curing methods, surface finishes and textures, environmental conditions, and craftsmanship (Hospodarova *et al.,* 2016).

It was estimated that the production of cement contributes to 5–7 % of CO_2 emission yearly, where for each ton of cement produced, 0.55 tons of CO_2 is released due to the decomposition of calcium carbonate during the clinkering process and 0.4 tons of CO_2 is produced by the combustion of carbon-fuel for the energy production (Lee *et al.,*2019).

Concrete used to be gray, it used to be all gray (De Magalhaes *et al.,* 2020). But of recent, engineers, architects and designers use coloured cement as a tool to incorporate an element of innovation, differentiation and exclusivity to their projects (Hospodarova *et al.,* 2016). Thanks to colour the possibilities in architecture design have been expanded (Belykh *et al.,* 2018).

Furthermore, cementitious products manufacturers are aware of the importance of adding colour to cement, as it makes it possible to give an added value to their products and have a degree of differentiation from their competition (De Magalhaes *et al.,* 2020).This increase in the use of coloured construction materials has been possible thanks to new developments in the incorporation and handling of pigments in recent years (Gholampour 2017).

A Pigment is a matter that alters or enhances the colour of any object as an outcome of absorption. Pigmentation in bricks is very vital in the sense that it changes dull objects into eye catching marketable product (Christie, 2015). Iron Oxides pigment are matters that are non-toxic and can resist harsh weather (Deng *et al.*, 2020), there are two types of iron oxides pigment which have been used for concrete colouration, they are Natural and Synthetic Iron Oxide.

It's important to understand that Iron Oxide is a pigment, a crystallized powder, and is it not a dye or paint itself (Souza, 2020). Only being added to the cement or other premixes, Iron Oxide can give colour to the final product (Hospodarova *et al.*, 2016). Iron oxide pigments produce rich and durable colours in concrete and other cementitious materials, because it has the following advantages: Good weather ability, good light fastness, excellent resistance in alkaline environment of concrete, tinting strength and fine dispersion inbuilt in the concrete (Hatami *et al.*, 2017). This means iron oxide pigments do not deteriorate over time experiencing various irritations such as rains, extreme UV exposure, and shoe-surface friction (Yun *et al.*, 2019). Also, Iron oxide does not just dissolve in a mixture, it coats the surface of cement particles and does not change the colour of sand or other aggregates in the mixture (Hatami *et al.*, 2021), which leads to two outcomes, adding more pigment to the cement will not lead to a more intensive colour, it will worsen the properties of the final product and Other aggregates may influence the final colour (Hatami *et al.*, 2021).

Key components that every manufacturer should have in mind when using iron oxide pigments to colour concrete:

1. The quality of the Pigment
2. Water to Cement Ration
3. Cement Content
4. Aggregate colour.

There are other pigments for colouration such as Chromium oxide, Cobalt oxide, Titanium Oxide, Manganese dioxide but are highly expensive and are purchased abroad unlike iron oxide which is available in building material markets in Nigeria and is affordable. The role of pigment in cement is to beautify the product. Pigments are added to cement to give it a specific colour or to enhance its appearance (Souza, 2020). Pigments can also improve the durability, UV resistance of the cement, making it less likely to fade or discolor over time. The primary role of pigment in cement is to colour or tint the cement, making it visually appealing and more aesthetically pleasing (Hatami *et al.*, 2021).

This research work is carried out to produce pigmented colour cement using Iron Oxide (Fe_2O_3) as the pigment, for direct production of coloured cement and reduce cost of purchasing different materials for the same purpose.

2 Materials and Methods

2.1 Materials: Chemicals used for this research were of analytical grade. The solvent used for this research was distilled water obtained from Panlac Chemical Ltd. Sodium Hydroxide (NaOH) was used as the precipitate. Iron Oxide (Fe_2O_3) pigments and Dangote Cement of 42.5R grade was obtained from Dei Dei International building market Abuja, Nigeria.

2.2 Production of Red Pigment: Iron Oxide pigments were used for the production process, 15g of ferrous sulphate was crushed and purified to remove impurities and to achieve consistent composition. A solution of the iron salt is prepared and reacted with a precipitating agent Sodium Hydroxide (NaOH) in the presence of an oxidizing agent, such as hydrogen peroxide or sodium chlorate. This causes the iron to form a solid precipitate of ferric hydroxide. The ferric hydroxide precipitate is allowed to age, typically for several days, to allow the particles to grow in size and develop the desired red colour. The aged precipitate is then washed several times with water to remove any impurities or remaining precipitating agent. The washed precipitate is dried, either by air-drying or by heating in an oven. The dried precipitate is then calcined at a temperature, between 800 - 900°C, to convert the ferric hydroxide to red iron oxide. The calcined red iron oxide is ground into a fine powder.

2.3 Production of Pigmented Colour Cement:

The first sample was produced using 25kg weight of the red pigment as, 1kg of Iron Oxide was measured, weighed in a scale and mixed with 50kg of Dangote Blocmaster cement in a pre-blending concrete mixer and allowed to mix thoroughly. It was observed that the Pre-blended cement colour was not sharp. In the second trial mix, an addition of 1kg extra Iron Oxide (Red) pigment into the already mixed cement, gave a change we desire, but not sharp enough. A third trial was conducted by addition of another 1kg extra Iron Oxide (Red) into the already mixed cement which gave the sharpest and desired colour.

2.4 Inter-lock/Kerbs Production Using the New Product (Pigmented Cement):

Production of Interlocks and kerbs stones were carried out by separating two and a half wheelbarrow of stone dust to 50kg of pigmented cement for four sets of samples (3kg red). Hand mixing method was done and to be turned 3 times to ensure uniform mixing is achieved, clean water was added to each sample mix and turned until slightly soluble mould is achieved and placed in interlock containers for surfacing and drying to take place as shown in Figures 2.1 and 2.2, this was done for each set of samples (Nesreen *et al.*, 2015). The inter-lock / kerbs production was allowed to dry and monitored for 3 days (as standard). The inter-locks produced from the pigmented cement shown in Figure 2.3 were collected for further testing and laboratory analysis.



Figure 2.1: Mixing of Red Pigment for Interlock Production



Figure 2.2: Drying of Interlock from Red pigment



Figure 2.3: General overview of Produced Interlock, Kerbs

Research Through Innovation

3. Results and Discussions

In FTIR spectroscopy, the wavenumber and intensity of the peaks can provide information about the chemical bonds present in the sample. The wavenumber corresponds to the energy of the light absorbed, which is characteristic of a specific type of bond. The intensity of the peak can give an indication of the amount of that bond present. Recent research has explored its applications in quantifying hydration products, such as calcium silicate hydrate (C – S - H) gels (Chen *et al.*, 2021). This makes it possible to comprehend cement hydration kinetics better.

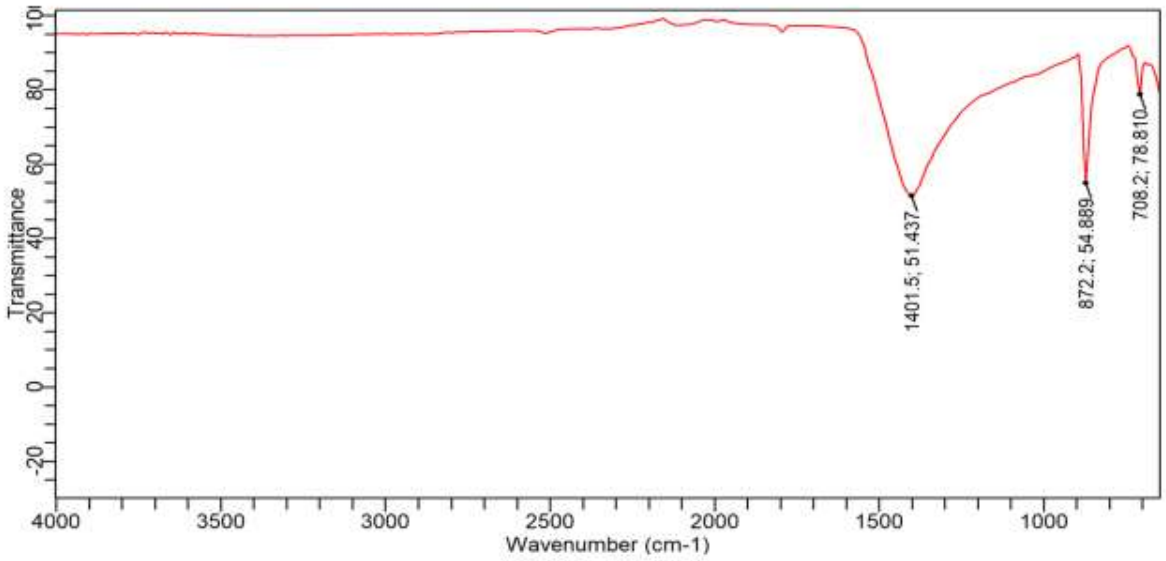


Figure 3.1: Ftir spectrum of raw red pigment

- Peak 1 at 708.19419 cm-1 could correspond to the Fe-O stretching vibrations in iron oxide.
- Peak 2 at 872.19705 cm-1 might be related to the bending modes of water (H_2O) or hydroxyl groups (OH-) present in the sample.
- Peak 3 at 1401.47903 cm-1 could be associated with carbonate (CO_3^{2-}) groups, indicating the presence of carbonates in the sample.

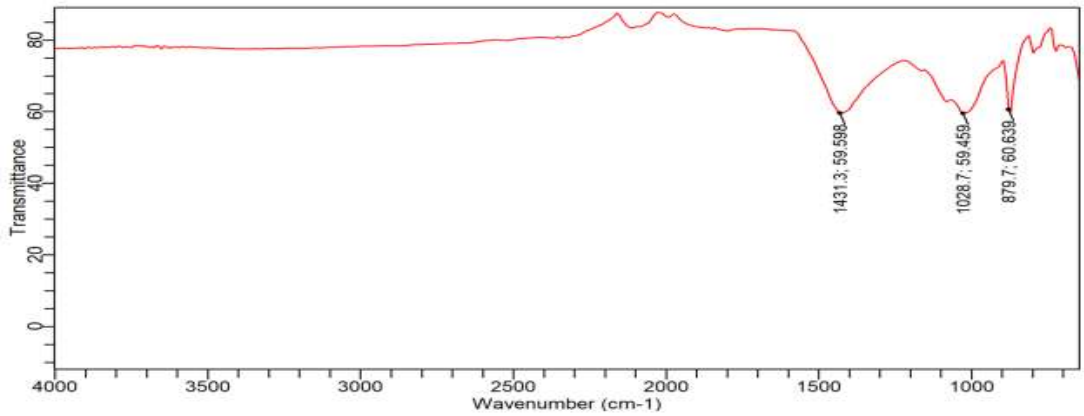


Figure 3.2: Ftir spectrum of raw produced red pigment

- Peak 1 at 879.65173 cm-1: This could be associated with the C-H bending vibrations in organic compounds. If the iron oxide powder is organic or has organic additives, this peak could be related to those components.
- Peak 2 at 1028.74524 cm-1: This wavenumber is often associated with C-O stretching vibrations in ethers and alcohol groups. If the iron oxide powder contains these functional groups, this peak could be indicative of them.
- Peak 3 at 1431.29773 cm-1: This could be associated with C-H bending vibrations in the plane of the molecule, often seen in alkenes and aromatics.

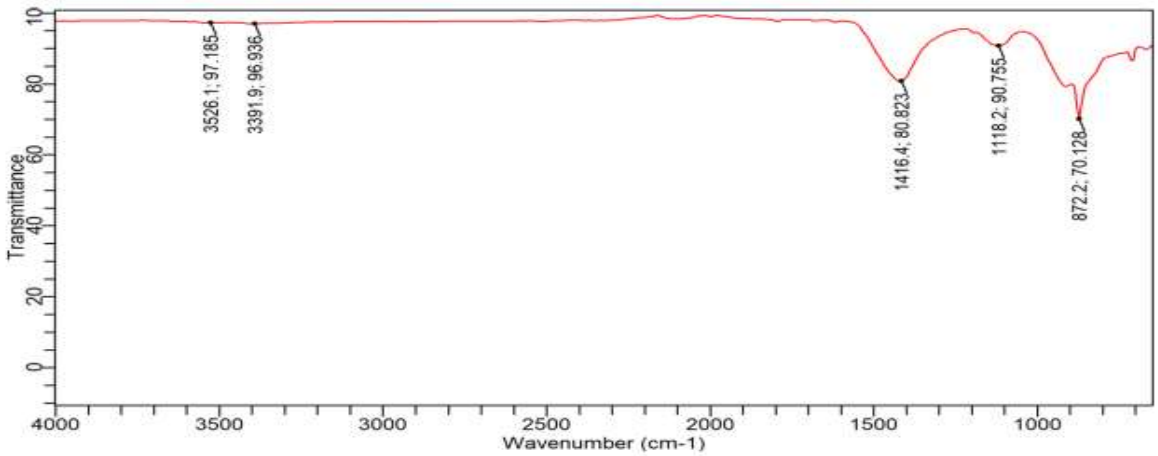


Figure 3.3: Ftir spectrum of portland cement

- Peak 1 at 872.19705 cm-1: This could be associated with the bending modes of water (H_2O) or hydroxyl groups (OH-) present in the sample.
- Peak 2 at 1118.20135 cm-1: This wavenumber is often associated with Si-O stretching vibrations in silicates, which are a major component of Portland cement.
- Peak 3 at 1416.38838 cm-1: This could be associated with carbonate (CO_3^{2-}) groups, indicating the presence of carbonates in the cement.
- Peak 4 at 3391.81143 cm-1 and Peak 5 at 3526.06159 cm-1: These peaks in the high wavenumber region are typically due to O-H stretching vibrations, indicating the presence of hydroxyl groups or water in the sample.

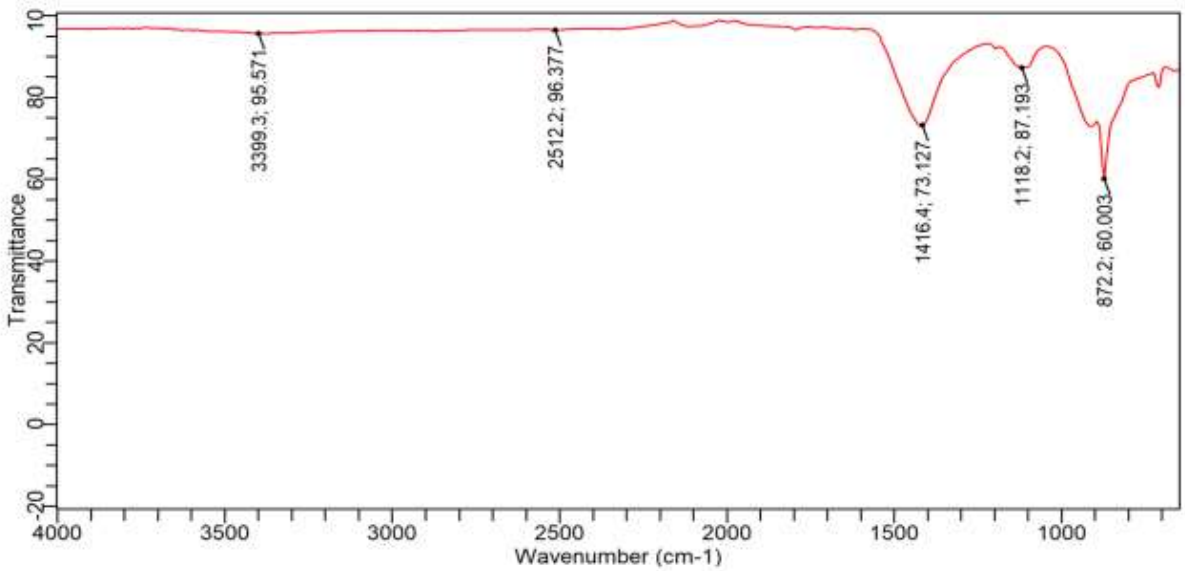


Figure 3.4: Ftir spectrum of red pigment cement

- Peak 1 at 872.19705 cm-1: This could be associated with the bending modes of water (H_2O) or hydroxyl groups (OH-) present in the sample.
- Peak 2 at 1118.20135 cm-1: This wavenumber is often associated with Si-O stretching vibrations in silicates, which are a major component of Portland cement.
- Peak 3 at 1416.38838 cm-1: This could be associated with carbonate (CO_3^{2-}) groups, indicating the presence of carbonates in the cement.
- Peak 4 at 2512.22570 cm-1: This peak is associated with the combined iron oxide groups.
- Peak 5 at 3399.33211 cm-1: This peak in the high wavenumber region is typically due to O-H stretching vibrations, indicating the presence of hydroxyl groups or water in the sample.

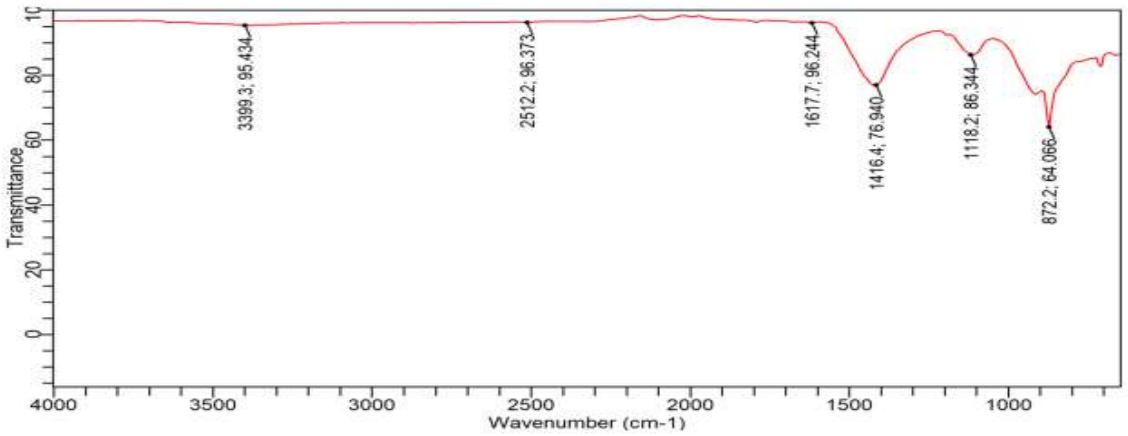


Figure 3.5: Ftir spectrum of produced red pigment cement

- Peak 1 at 872.19705 cm-1: This could be associated with the bending modes of water (H_2O) or hydroxyl groups (OH-) present in the sample.
- Peak 2 at 1118.20135 cm-1: This wavenumber is often associated with Si-O stretching vibrations in silicates, which could be present in the sample.
- Peak 3 at 1416.38838 cm-1: This could be associated with carbonate (CO_3^{2-}) groups, indicating the presence of carbonates in the sample.
- Peak 4 at 1617.66462 cm-1: This peak could be associated with C=O stretching vibrations, indicating the presence of carbonyl groups in the sample.
- Peak 5 at 2512.22570 cm-1: This peak is colored iron pigment group.

The FTIR analysis carried out confirmed that the introduction of colour pigment was successful and no significant chemical changes were occurred when added to already existing Portland cement, and also the production of the produced red pigment had no changes as compared to the purchased pigments.

X-RAY DIFFRACTION (XRD):

The X-ray diffraction (XRD) pattern provides the chemical composition and crystalline structure of the mixes at the time of testing. So, results are directly related to the content of the hydration phases.

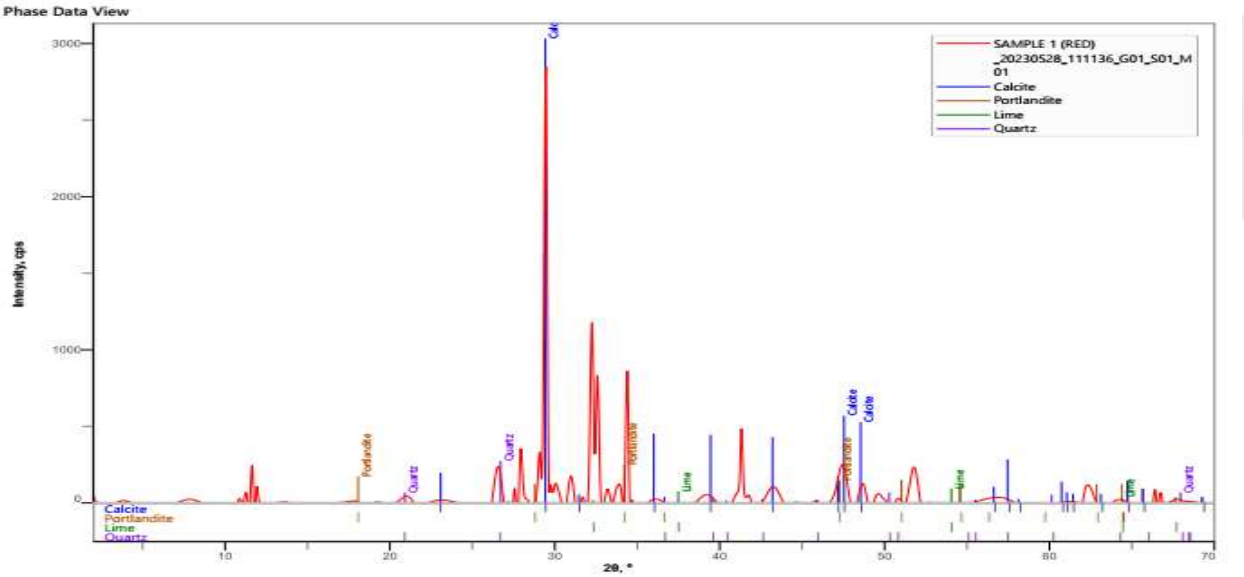
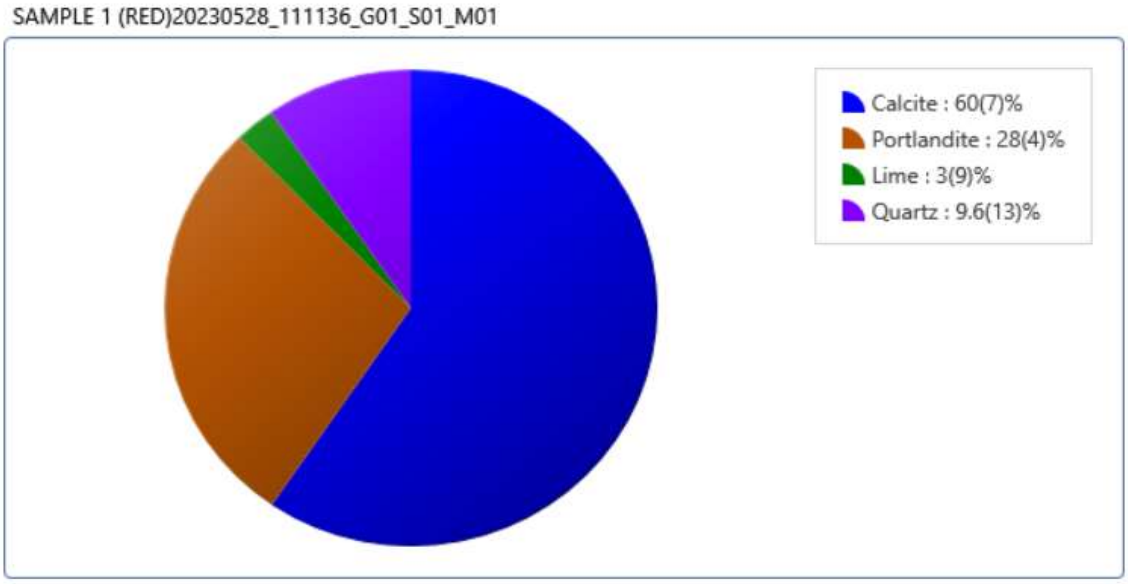


Figure 3.6: XRD PATTERN OF RED PIGMENTED CEMENT



PIE REPRESENTATION OF COMPONENTS FOR RED PIGMENT CEMENT.

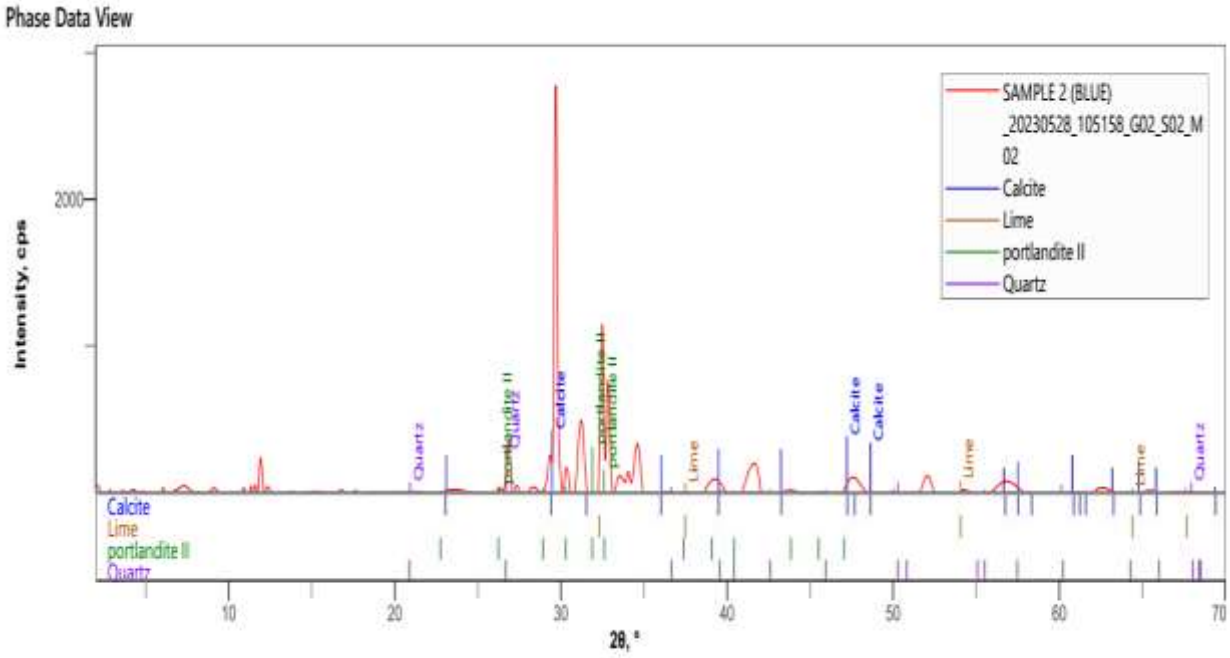
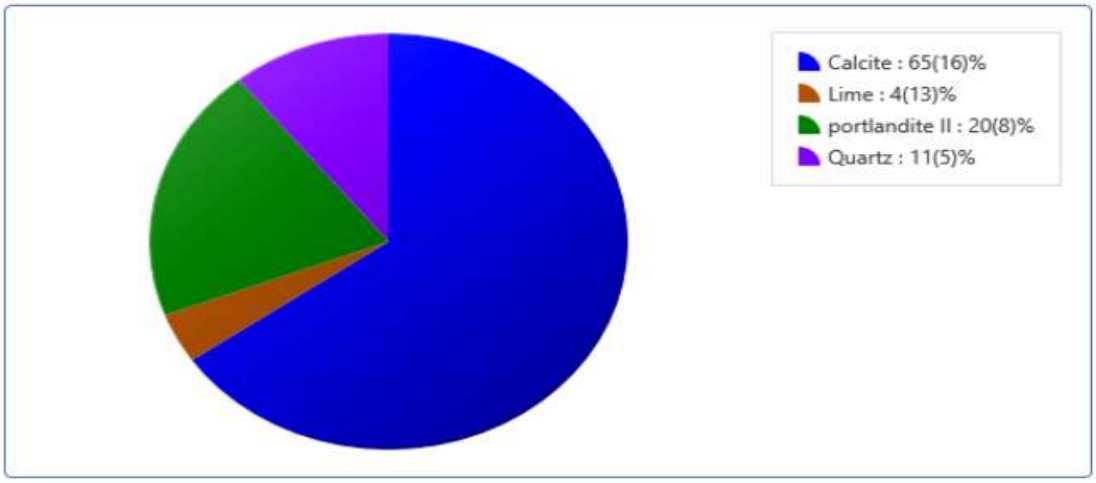


Figure 3.7: XRD PATTERN OF PRODUCED RED PIGMENTED CEMENT



PIE REPRESENTATION OF COMPONENTS FOR RED PIGMENT CEMENT.

Figures 3.6 and 3.7 present the results obtained from the red pigmented cement and the produced red pigment cement after the age of 3 and 28 days curing. The main components identified are: Calcite, Lime, Portlandite and Quartz which are shared in their various proportions in the pie chart results.

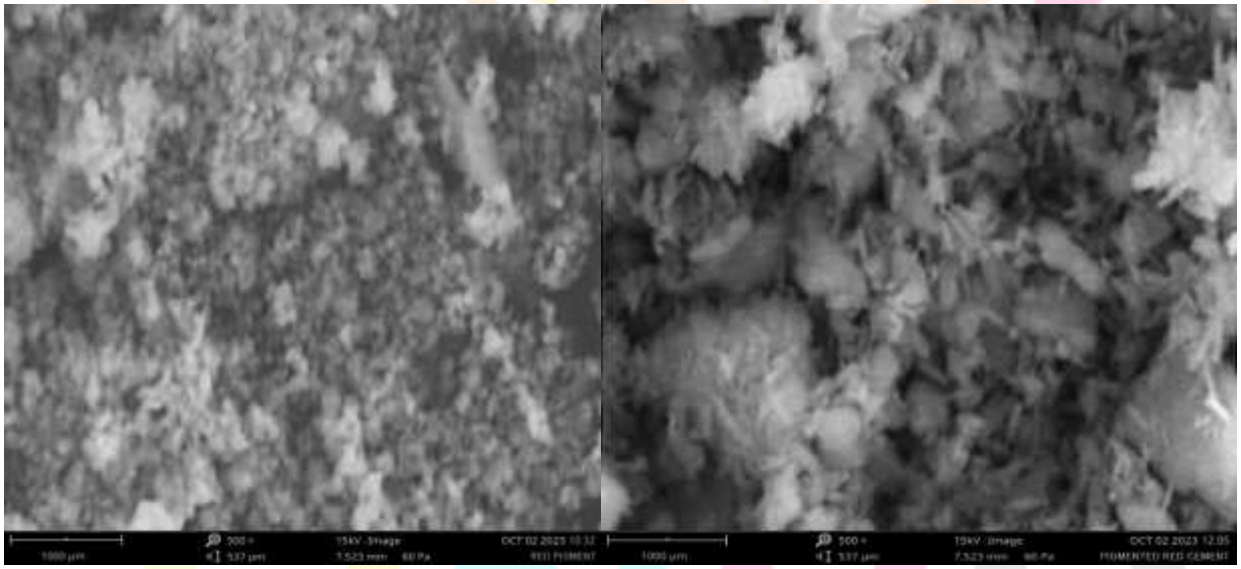


Figure 3.8: SEM micrograph of red pigment Figure 3.9: SEM micrograph of pigmented red cement

The scanning electron microscope (SEM) results for the red pigment and pigmented cement reveal distinct differences in their particle arrangement and overall structure.

In the case of the red pigment alone (Figure 3.8), the SEM analysis indicates a densely compacted particle structure. This suggests that the red pigment particles are closely packed together, forming a uniform mass. The dense arrangement of particles can be attributed to several factors, such as the size, shape, and surface properties of the pigment particles themselves.

On the other hand, when examining the pigmented cement (Figure 3.9), the SEM analysis showcases a bit more loose and semi-spherical structure. The presence of the red pigment within the Portland cement matrix can introduce variations in particle distribution and packing. The addition of the pigment particles caused the disruption of the original arrangement of the cement particles, leading to a less compacted structure.

Table 1: Compressive strength test for 3 days aging for produced red pigment kerb.

S/No	Sample ID	Concrete Age	Weight in Air (kg)	Volume m^3	Density (kg/m^3)	Load (kN)	Compressive Strength N/mm^2	Av. Compressive Strength N/mm^2
1	A	3 Days	3.560	0.001728	2060	174.078	6.063	5.885
2	B		3.545	0.001728	2052	172.051	5.992	
3	C		3.640	0.001728	2106	162.544	5.601	

Table 2: Compressive strength test for 3 days aging for red pigment kerb

S/No	Sample ID	Concrete Age	Weight in Air (kg)	Volume m^3	Density (kg/m^3)	Load (kN)	Compressive Strength N/mm^2	Av. Compressive Strength N/mm^2
1	A	3 Days	3.815	0.001728	2208	174.539	6.079	5.878
2	B		3.440	0.001728	1991	159.567	5.557	
3	C		3.675	0.001728	2127	172.252	5.999	

Table 3: Compressive strength test for 28 days aging for red pigment kerb

S/No	Sample ID	Concrete Age	Weight in Air (kg)	Volume m^3	Density (kg/m^3)	Load (kN)	Compressive Strength N/mm^2	Av. Compressive Strength N/mm^2
1	A	28 Days	3.855	0.001728	2231	181.451	6.585	6.558
2	B		3.380	0.001728	1956	177.660	6.449	
3	C		3.380	0.001728	1956	182.944	6.640	

Table 4: Compressive strength test for 28 days aging for produced red pigment kerb

S/No	Sample ID	Concrete Age	Weight in Air (kg)	Volume m^3	Density (kg/m^3)	Load (kN)	Compressive Strength N/mm^2	Av. Compressive Strength N/mm^2
1	A	28 Days	3.875	0.001728	2242	188.527	6.843	6.611
2	B		3.890	0.001728	2251	177.224	6.433	
3	C		3.535	0.001728	2046	180.610	6.556	

The compressive strengths were determined in accordance with the EN 196-1:2016. Prismatic specimens (195 × 195 × 98 mm) were cast, compacted and cured in a control chamber at a temperature of 20 ± 2ºC and humidity of 98 ± 1 % until the age of testing (3 and 28 days). At each age, the compressive strength achieved are the average of 3 and 28 days tests, respectively.

Table 5: Water absorption test for red pigment (A) and produced red pigment cement (A')

	A	A'
Mass of saturated surface dry (SSD) (g)	3740	3790
Dry Mass at Constant Temperature (g)	3530	3510
Dry Mass (g)	210.00	280.00
Water Absorption (%)	5.95	7.98
Average (%)	6.96	

Water absorption and porosity were calculated following the standard BS 1881-122:2011. Then, three kerbs of (195 × 195 × 98 mm) were prepared for each mix. After a curing of 28 days, samples were dried in an oven at 105 ± 5°C for 48hrs. After that, samples were left to cool in a desiccator for 24 hours and weighted to obtain the dry mass (g). The samples were immersed in water for 24 hours. Then, the samples were weighted to obtain the saturated surface dry (g). Lastly, water absorption (WA) and the porosity (P) were calculated with Eqns 1&2. ρ_w is the density of water at 20 ◦C (998.2 kg/m^3).

$$WA(\%) = \frac{Ms - Md}{Md} \quad (1)$$

$$P(\%) = \frac{\frac{Ms - Md}{\rho_w}}{Vs} \quad (2)$$

Table 6: Physical test on pigmented cement

ITEM	SPEC. LIMIT	TEST RESULT
Blaine Fineness (m²/kg) (c204)	200-400	281
Standard Consistency	26-33	28
Initial setting time (mins)	≥45	152
Final setting time (mins)	>600	241
Loss of Ignition	≤4%	1.37%

The Blaine fineness of pigmented cement was determined to be 281 m^2/kg, falling within the specified range of 200 - 400 m^2/kg. This result indicates satisfactory particle fineness, suggesting adequate surface area for chemical reactions during hydration. The standard consistency of the pigmented cement was measured at 28%, within the acceptable range of 26-33%. This finding implies that the cement paste exhibited appropriate flow and workability characteristics, meeting the desired consistency standards. The initial setting time of pigmented cement was found to be 152 minutes, surpassing the minimum requirement of 45 minutes. This extended setting time suggests that the cement maintains a workable consistency for an extended duration, facilitating construction processes. The final setting time was determined to be 241 minutes, well below the maximum limit of 600 minutes. This indicates that the pigmented cement achieves its final set within a reasonable time frame, contributing to the overall efficiency and reliability of construction applications.

4. Conclusions

In conclusion, the addition of Fe_2O_3 pigment to cement has been found to have a positive effect on the properties of coloured cement for concrete works. The study have reported minimal effect on compressive strength, while enhancing durability, colour stability, and workability when Fe_2O_3 pigment is added to cement, from the results obtained the introduction Fe_2O_3 has no negative impact of the of changes in the concrete strength, soundness, fineness of the pigmented cement, however the properties of coloured cement depend the type of pigment used and the composition of the pigment. The optimal dosage of Fe_2O_3 pigment ranges from 4% to 6% by weight of cement, depending on the specific application.

Conflict of Interest

We hereby declare that there is no conflict of interest regarding the research presented in the manuscript titled "[Investigation on the Effect of Iron Oxide (Fe_2O_3) Pigment on the Production of Coloured Cement for Concrete Works]."

5. References

1. Belykh, I., Sopov, V., Butska, L., Pershina, L., & Makarenko O. (2018), Predicting the strength and maturity of hardening concrete, in: MATEC Web of Conferences.

2. Chen, X., Guo, H., Wang, S., & Zhang, Z. (2021). Quantitative characterization of calcium silicate hydrate gels in cementitious materials by FTIR spectroscopy. *Cement and Concrete Research*, 140, 106-299

3. Christie, R. Colour Chemistry, Royal Society of Chemistry, Cambridge, (2015).

4. Deng, X., Li, J., Lu, Z., Chen, J., Luo, K., Nui, Y. (2020) Effect of hydrated lime on structures and properties of decorative rendering mortar, *Constr. Build. Mater*. 7(4). 58-68.

5. Gholampour, T. O. (2017), Performance of sustainable concretes containing very high-volume class-F fly ash and ground granulated blast furnace slag, *J. Clean. Prod.* 162.1407–1417.

6. L. Hatami, M. Jamshidi (2021), Effects of type and duration of pigment milling on mechanical and colorimetric properties of coloured self-compacting mortars, J. Build.Eng. 35

7. Heerah, M. Z., Galobardes, I., & Dawson, G. (2021). Characterisation and control of cementitious mixes with colour pigment admixtures. *Case Studies in Construction Materials*, *15*, e00571. https://doi.org/10.1016/j.cscm.2021.e00571

8. Hospodarova, V., Junak, J., & Stevulova, N. (2016). Color pigments in concrete and their properties, *Int. J. Eng. Inf. Sci.* 10 (3), 143–151.

9. Lee, J. D., & Ahmad, S. (2019). Natural and synthetic iron oxide pigments: Production, properties, and applications. *Inorganic Chemistry Frontiers*, 6(2), 295-311.

10. Nesreen E., Kamal G. S., Hussein, N. A. (2015). Advantages and Disadvantages of Coloured Concrete in Structural Engineering. *PSERJ* 19, 1-10.

11. Souza, A. T., Barbosa, T. F., & Riccio, L.A. (2020). Effects of limestone powder substitution on mechanical properties and durability of slender precast components of structural mortar, *J. Mater. Res. Technol.* 9 (1) 847–856.

12. Yun, H. D., Lee, J. W., Jang, Y. I., Jang, S. J., & Choi W. (2019), Microstructure and mechanical properties of cement mortar containing phase change materials, *Appl. Sci.* 9, 101-110.

13. S. P. Dunuweera and R. M. G. Rajapakse, "Cement types,composition, uses, environmental impact and possible solutions," in Proceedings of the 28th International Symposium on Transport Phenomena, Peradeniya, Sri Lanka, September 2017.