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Color Mismatch in Compounding of Polycarbonate Composition: Processing Parameters and Pigment Dispersion Effects

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Abstract

The purpose of this research is to study the variations of the independent variable of processing conditions such as temp, speed, and feed rate that affect the dependent responses for consistent output color (L*, a*, b*, dE*). In this study, the compounded material prepared on an intermeshing twin-screw extruder (TSE) and injection molded to evaluate their effect on the color stability, rheology, and dispersion of the polycarbonate resins. The focus was continued to the interaction of the speed which correlates to the dispersion and color changes. The interaction relationship between tristimulus color values (dL*, da*, db*, dE*) and processing parameters showed that the minimum color difference throughout the experiment was 68.42 for L*, 1.47 for a*, 15.35 for b*, and 0.34 for dE* respectively.The color difference values (dE*) decrease significantly with the increase of the temperature and feed rates.

1. Introduction

Manufacture the best color; it is essential for the design and the control of most manufactured colored materials to take into consideration the mastery of the material science including physics, chemistry, and sensibility [1,2,3,4]. In the 21st century, color science is more significant than ever. Manufacturing plastic with a favorite color involves merely adding to one another, one or more polymers; nevertheless reaching the correct color in the first shot is a challenge. Apart from pigment formulations, the color properties of plastic are directly affected by the processing conditions for material compounding during the extrusion system. Data mining, in which historical data are analyzed to select formulations (PC compound grades, e.g.G-3), pigments, e.g. (red pigment), processing parameters (temp, rpm, feed rate) were made color mismatches. Blending mixer, co-rotating intermeshing- twin screw extruder and injection molding were performed. General factorial design, box Behnken design (BBD) with the threelevels of the experiment were conducted [5,6,7,8]. ANOVA of the obtained experimental data was utilized to study the effects of these processing parameters. The regression model was generated based on the ANOVA to predict the optimal matching color. Producing the right to with minimal wastage has been a big challenge [9]. The Polymer color varies with changes in the light, the object, and the observer sources [10, 11, 13]. The effects of screw geometry and operating conditions on dispersion have also been studied by Philipp et al. [12]. The results show that the controlling the particle size and distribution of a yellow pigment, PY62 directly affects properties such as film, transparency, color development, extruder pressure build and processing time [13,14]. The color variation is due to degradation, temperature, particle size and colorant [15]. Screw speeds have no apparent effect on the kneading block performance [16]. Shear forces that occur in the extruder are in a good determinant of the level of mixing with a different polymer [17]. The high shear rates, processing temperatures, and processing pressures involved in the manufacturing processes. The increasing speed (screw speed) will enhance the longitudinal distribution [18, 19]. Processing affects the rheological properties and dispersion ability of pigments in the resin [20, 21, 22, 23, 24]. The colors are metallic products because they do not exhibit exact and uniform size or range of sizes [25]. Narrower particle size distributions are superior for Chroma Scattering, and absorption is functions of particle size. The control of properties denotes associated with particle size and morphological structure. Characteristically, narrower particle size distribution shows a cleaner effect and wider provides greater hiding power. Therefore, the shapes offer different levels of reflectance and opacity [26]. To obtain primary particle size, it is necessary to have a smaller particle which has a higher surface area and consequently stronger the color [27, 28, 29, 30]. The Systematic progress may broadly denote divided into two types of reducing the particle size, shear and collision. The best superiority of the dispersion grade depends on the characteristic of the concentration of Pigment, volume, time spent, rotation speed, energy input, and temperature [31,32].The dispersion degree depends on the energy use in, i.e., speed and the residence time in the chamber. The intensity of scattered light by a particle depends on its molecular weight, molecular size and geometrical shape [33].

This research presents experimental observations and statistical analysis to investigate scientific reasons for color mismatches in compounded plastics. Material processing issues were observed. Different methodologies were developed and applied to study and improve the understanding of color matching, color stability and consistency of compounded plastic materials, to minimize wastage. The first used Data Mining, in which historical data were analyzed to select formulations, particularly for pigments and polycarbonate blends of compounded plastics, which were known to suffer from the color mismatch.

Two data mining techniques were utilized. One was online analytical processing (OLAP), and the other was a decision tree classifier (DTC). The DTC will be used for exploring parameters most likely to effect color failure problems with compounded plastics. Therefore, Online Analytical Processing (OLAP), DTC and neural network modeling (NNM) are sorting through large data sets approaches have been executed to discover such parameter [34,35,36,37,38,39]. The second data mining used the parametric study to investigate issues in the dispersion of pigments by determining and analyzing the effects of processing parameters on color. Finally, the third method studied the effects of rheology.

The results indicate that the variation in temperature significantly affected rheological properties and hence color deviations. The fourth method looked at effects of processing parameters on dispersion, pigment size distribution (PSD) and morphology of pigments in polycarbonate compounds

The goal of this research is to study the impact of processing conditions, rheology, and dispersion. Experiments were produced regarding three different processing parameters (Temp, feed rate, Screw Speed), while keeping the other parameters fixed at (255 °C, 25kg/hr., 750rpm). In our earlier work [46, 47] found that the achieved results of the deviation in polycarbonate content and temperature crucially effect on rheological characteristics and the obtained viscosity data were correlated to dispersion and color changes. In this study, we use the same translucent PC compound grade at three level design, each of the runs was replicated three times to allow estimation of experimental error or trend analysis (GT). This work investigated the variety of independent of processing parameters such as temp, screw speed, and feed rate are affected the dependent responses for consistent output color (L*, a*, b*, dE*). Focus extended on the speed, correlated to dispersion and color changes to evaluate their effect on the color stability, rheology and dispersion of the translucent compounded plastic grade -3 of polycarbonate (PC) achieve minimum deviation of color differences.

2. Experimental details

2.1 Raw materials

A commercial Ordinary Portland Cement (OPC) (43 grade, specific gravity 3.15, Blaine's Fineness 2630 cm^2/g) complying with ASTM C 150 standards was used. The Class C Fly Ash (RFA) obtained from Neyveli Lignite Corporation; Neyveli, Tamil Nadu was utilized for the study. The RFA was ground for 2 hours to reduce its size and EFA is produced. Distilled water having the pH value as seven was used for mortar preparation and casting of specimens. Conforming to IS 650, three different grades (Grade I, II and III) of standard Ennore sand has been used as the fine aggregate for preparing standard mortar samples. Powdered type Nano-silica was in light rose color having a density of 2.2 g/cm^3 was utilized in this study. Conplast SP 340 which was based on sulfonated naphthalene polymers was used as superplasticizers in the percentage of 1% to 1.3%. For the Acid resistance test three types of acids were used at the 0.1 molarity of Sulfuric acid (H_2SO_4), Hydrochloric acid (HCL) and Nitric acid (HNO_3) respectively.

2.2 Specimen Preparation and Testing

The preliminary tests were conducted to determine the water to binder ratio. Firstly, normal consistency for cement was carried out in accordance to IS 4031-Part 4, and the water requirement for the mortar was identified. Secondly, with the determined consistency, the water to binder ratio for the cement mortar was optimized using flow table test. The flow table test was performed conforming to IS 5512, and the water to binder ratio was found to be workable at 0.55. [16, 17]

The cement pastes incorporating Nano silica (NS) and Fly Ash were prepared at Standard Consistency using a planetary mixer. For all the types of paste mix design that adopted was confirming to IS 10262-2009. The adopted mix ratio for the mortar mix was 1: 2.75. The water binding ratio's taken for mix combinations with and without 1% superplasticizers were 0.55 and 0.4 respectively. For compression strength test and Sorptivity test, the casting of 50mm size cubes was done. After 24 hours, specimens were de-moulded and immersed in the water tank for curing up to the testing age. Compression strength of the sample were determined as per ASTM C109 code specifications at 7th, 14th, 28th and 56th days of curing.

2.3 Microstructural Properties Analysis

The microstructures characteristics of OPC, RFA, EFA and NS sample used in this analysis by scanning electron microscope (SEM) in addition to the energy dispersive X-ray spectroscopy (EDX) to determine the elemental composition, morphology characters and texture[18].

3. Results

nsistency of fresh paste

Normal Consistency of the NS blended fly ash mortar samples was determined as per IS 4031-part 4-1988, and it was showed in Figure.1. It was observed that the consistency was more for the pozzolana mortar compare with the control mortar. Fineness is one of the major factors affecting the consistency of the mix. When comparing with the RFA mortar and EFA mortar, RFA mortar has more consistency at the same water content. It may be because, in EFA mortar, the number of particles that come into action is more due to its finer size. It was noticeable that when RFA and EFA were replaced with NS, the consistency was getting increased. It was because the particles are getting finer and the reaction increases and hence the consistency also increases [19-22]. Figure. 2 shows the flow table value of NS blended Fly ash mortar, and it was found that the EFA mortar shows more flow than RFA mortar. The rate of increase in flow with increase in the W/B ratio was lower if cement replaced with EFA and NS. But it was found that the all fly ash mortars had almost same workability when W/B ratio reaches 0.5.

It was noticed that the presence of carbon content in NS blended EFA mortar is more, which decelerate the pozzolanic activity. But the presence of Magnesium, Silica, and Iron might be the reason for the strength variation and increased durability in mortar specimens prepared using NS. Calcium content is high in EFA mortar, about 33.3% and was less in RFA mortar. That was the reason why EFA mortar shows better mechanical strength than the RFA mortar.

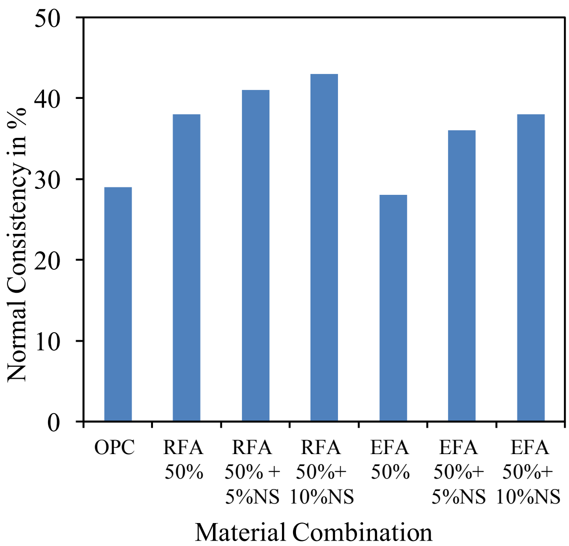

Figure 1: Normal Consistency results of NS samples

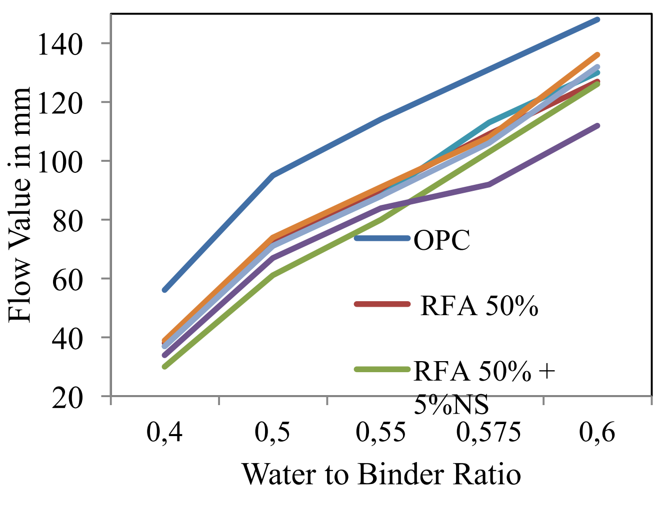

Figure 2: Flow table value of Blended Mortar Mix Ratio

3.2 Compressive Strength of Blended mortar

The graphical representation of compressive strength test result of mortar combination with and without superplasticizer shows in Figure 3 and Figure 4 respectively. It was found that replacement of cement with fly ash reduces the strength of the mortar. 50% replacement of cement with fly ash reduced 30% compressive strength in RFA mortar and 10% in EFA mortar [23-26].

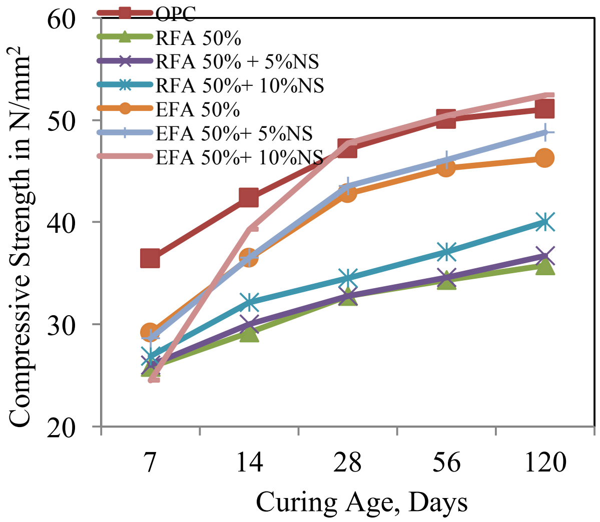

Figure 3: Compressive strength results of NS blended Fly ash mortar without SPL

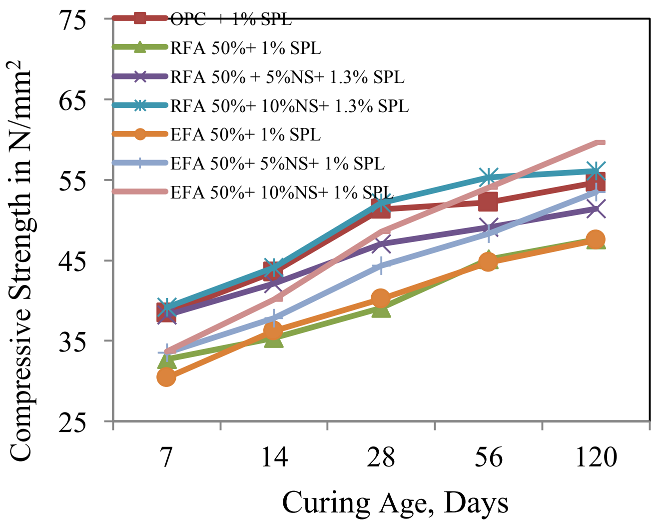

Figure 4: Compressive strength results of NS blended Fly ash mortar with SPL

When 5% fly ash is replaced with Nano-silica, the strength is gained in both RFA and EFA mortar. But still, it failed to meet the strength of control mortar. When 10% fly ash was replaced with NS, the mortar gains better compressive strength than the control mortar. The compressive strength of NS blended EFA mortar only have a compressive strength more or near the control mortar. NS combined EFA mortar exhibited better compressive strength than any other mortar samples. This can be explained by SEM analysis results. From the SEM image of NS mixed EFA specimen as shown in Figure. 4, it was clear that NS blended EFA sample had finer particles the EFA mortar. Hence they have more reactive surface area than the EFA mortar, and that was the reason better compressive strength.

3.3 Sorptivity Analysis Mortar specimens

Cubes of 50mm size were cast for all mortar samples to determine the rate of absorption of water by capillary suction. The rate of water absorption in 30 minutes of the period was studied. The Sorptivity values of mortars cast were compared in Figure. 5. It was observed that the NS blended Fly Ash mortar have fewer Sorptivity than the control mortar because of the fewer voids in the fly ash mortars. The finer particles in the fly ash mortars occupy thickly, and it will reduce the number of voids. The Sorptivity increased when the NS content increased in NS blended mortars. The reason was when the NS content has increased the diameter of the voids decreased into the very small dimension, the lower diameter voids; the greater would be the rate of capillary suction of water [27, 28]. Thus Capillary suction increases when the void diameter decreases, leading to increasing in the Sorptivity with the increase in NS content.

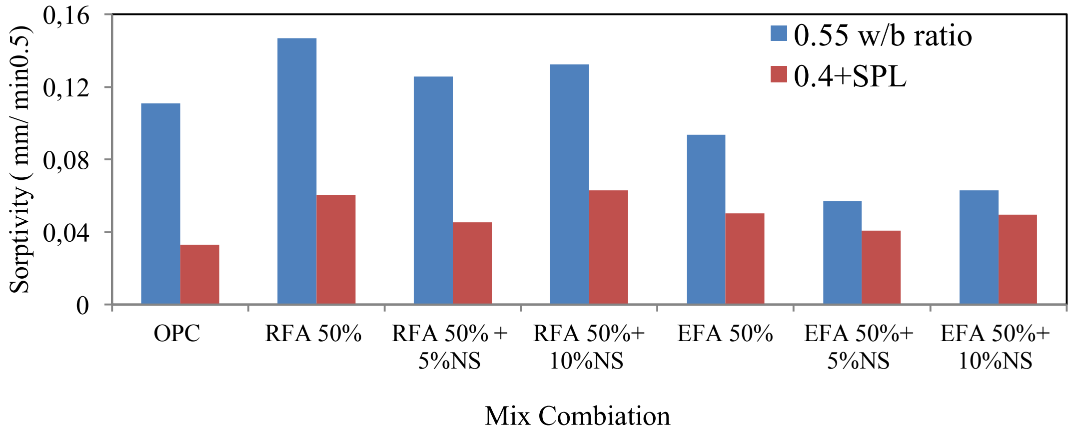

Figure 5: The comparison among the Sorptivity values of the mortar specimens

3.4 Acid Resistance Test

For testing the chemical resistance of the mortars, the variation in the compressive strength and weight change of the specimens immersed in acids were inspected. For compressive strength, 50 mm sized cubes were cast, cured for 28 days, immersed in HNO_3, HCl, and H_2SO_4 solutions separately for the next one month, dried and then tested. The 56th-day compressive strength of normally cured mortar specimen and acid immersed mortar specimens were showed in Figure 6 and Figure 7.

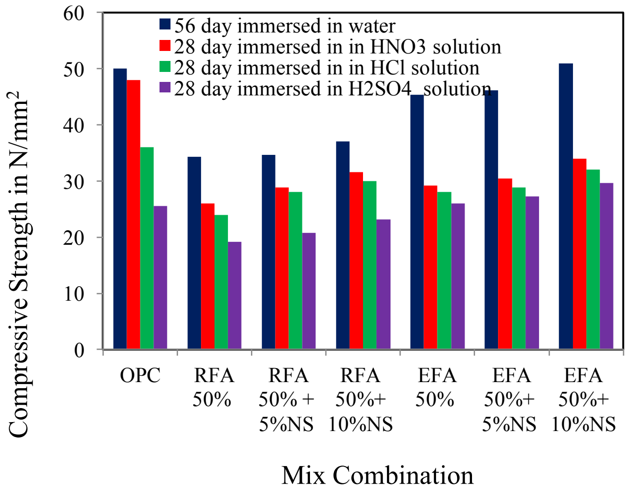

Figure 6: compressive strength of acid immersed mortar specimens (0.55 w/b ratio)

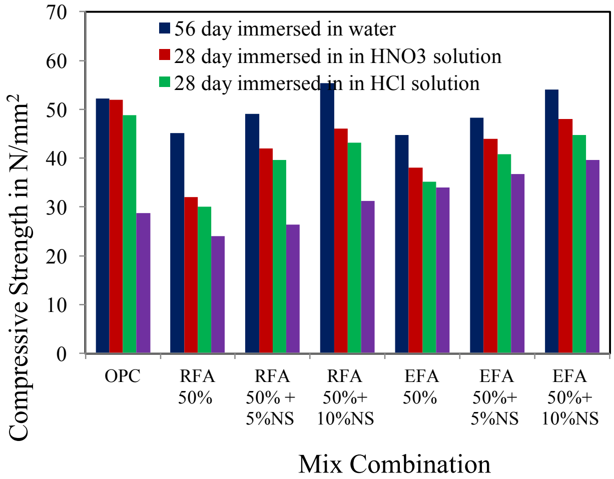

Figure 7: compressive strength of acid immersed mortar specimens (0.4+SPL w/b ratio)

It was observed that control mortar showed excellent resistance to HNO_3 and HCl solutions. But the strength of control mortars was decreased by 49% after immersing in H_2SO_4 solution. At the same time, NS blended EFA mortar immersed in H_2SO_4 solution are showing better acid resistance than the control mortar. It was also noticeable that the mortar cast with 0.40 W/B ratio showed exquisite acid resistance to all the acid solutions because of the low void presence in the specimens [23, 29-31]. The 10% SF blended EFA mortar shows better results, due to the low void presence in the mortar casted with 0.40 w/b ratio with shows better acid resistance than the control mortar. In this case the particle size was less compare to the control specimens.For investigating the loss of weight, the NS blended fly ash mortar specimens were immersed in the acid solution, and the loss in weight of each cube was monitored every week. Percentage of weight loss after four weeks of immersion in solution was shown in Figure. 8 and 9. From the Figures, it was observed that the Control mortar doesn't have any noticeable weight change after immersing the specimens in HNO_3 and HCl solution. But after immersing the control mortar in H_2SO_4 solution, considerable loss of weight was noticed. It was found that NS blended fly ash mortars had less percentage of weight loss than the control mortar when the specimens are immersed in H_2SO_4. But coming to the specimens immersed in HNO_3 and HCl solution other than control mortar all other mortar is facing an average of 0.6% weight loss.

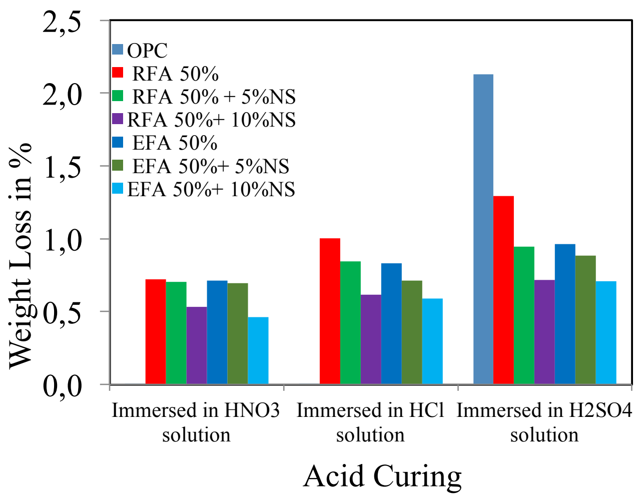

Figure 8: Weight loss of mortar immersed in acid solutions (0.55 w/b ratio)

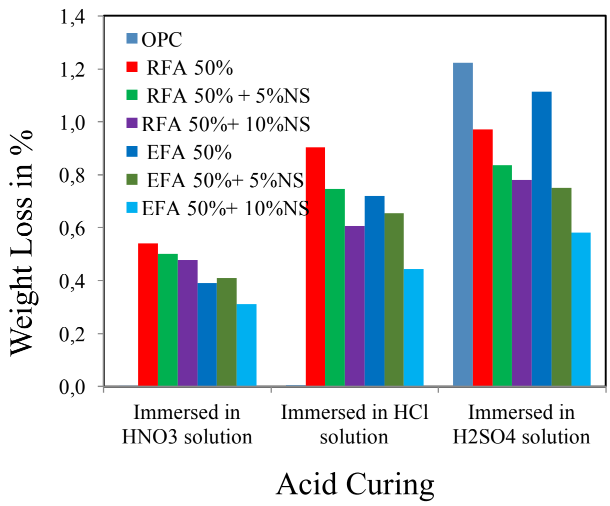

Figure 9: Weight loss of mortar immersed in acid solutions (0.4+SPL w/b ratio)

It was also noticed that the percentage weight loss of mortar specimens cast with 0.40 w/b ratio is lower than the percentage weight in loss of mortar specimens cast with 0.50 w/b ratio. It was observed that NS blended fly ash mortar is not losing significant weight comparing to other mortar specimens immersed in the acid solutions.

4. Microstructural Studies

4.1 Scanning Electron Microscopy (SEM) Analysis

SEM investigation was led to dissect the microstructure of the different mortar samples. Specimens at the 28th day were broken in to pieces and fragments were collected from the center of the sample. The fracture surface was observed by using SEM. All samples were layered with gold prior to the SEM analysis.

The shape of the particles was analyzed by using SEM analysis. Figure. 10,12 and 14 shows the SEM micrograph of OPC sample, NS blended RFA specimen, and NS blended EFA specimen respectively after 28 days curing. SEM analysis results revealed the shape of RFA samples was regular and spherical surface even though they were embedded in the paste for a long time. And EFA particles showed a non-spherical shape that was the reason why EFA mortar had less workability than RFA mortar [32,33].

From the SEM images, it was clear that NS blended EFA sample had finer particles the EFA mortar. Hence they have more reactive surface area than the EFA mortar, and that was the reason why NS combined EFA mortar exhibited better compressive strength than any other mortar samples.The nucleation effect and hydration reaction were enhanced by the incorporation of EFA. As a result, the specimen becomes more denser and homogenous than the specimen with the RFA.

4.2 Energy-Dispersive X-ray Spectroscopy (EDX) analysis

X-ray diffractograms of OPC sample, NS blended RFA specimen, and NS blended EFA specimen after 28 days curing were shown in Figure.11, 13 and 15 respectively. From Figure. 10, it was observed that Silica content is more in OPC mortar and that might be the reason that OPC mortar is showing decent mechanical and durability properties. It was noticeable that the presence of carbon content in NS blended EFA mortar is more, which decelerate the pozzolanic activity. But the presence of Magnesium, Silica and Iron might be the reason for the strength variation and increased durability in mortar specimens prepared using NS. Calcium content is high in EFA mortar, about 33.3% and is considerably less in RFA mortar. This might be the reason why EFA mortar shows better mechanical strength than the RFA mortar [34].

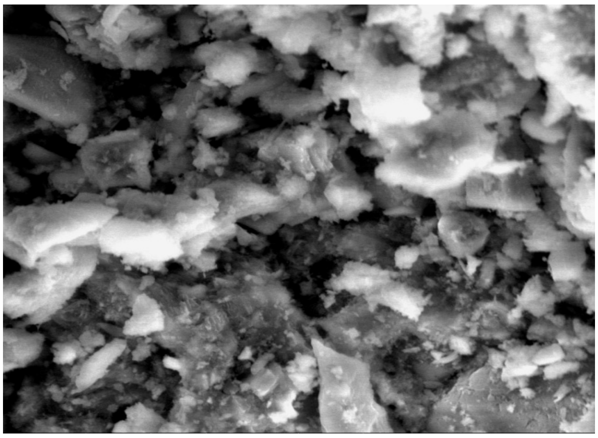

Figure 10: SEM Image of OPC mortar at 28 days curing

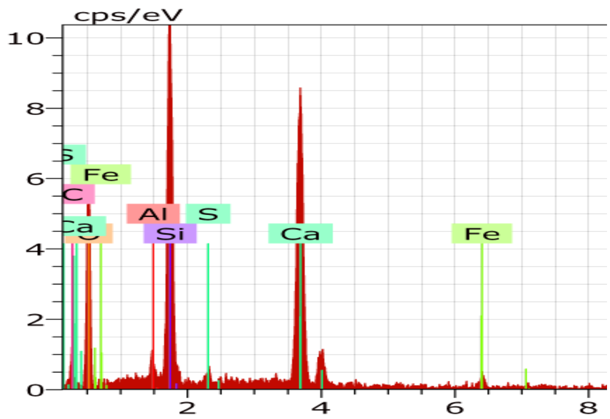

Figure 11: EDX analysis of OPC mortar at 28 days curing

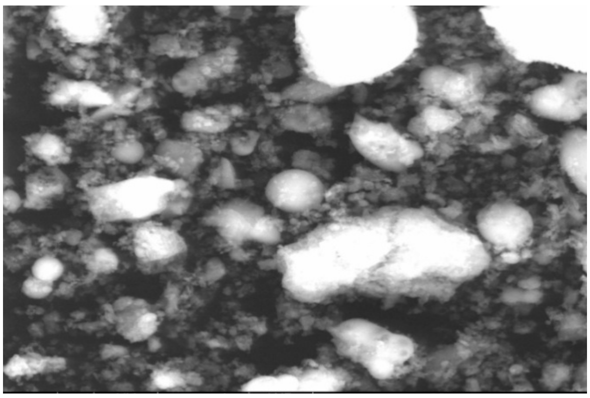

Figure 12: SEM Image of OPC replacement by RFA50%+10%NS with SPL at 28 days

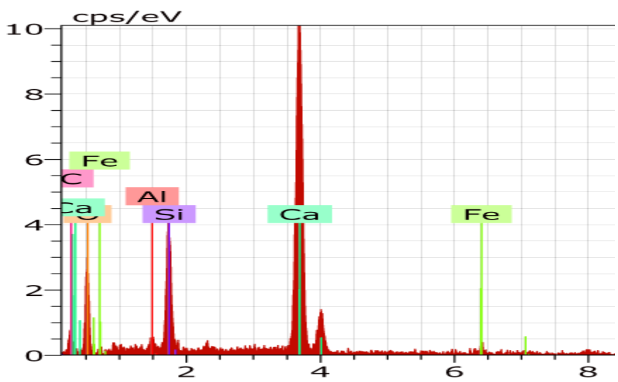

Figure 13: EDX analysis of RFA50%+10%NS with SPL at 28 days

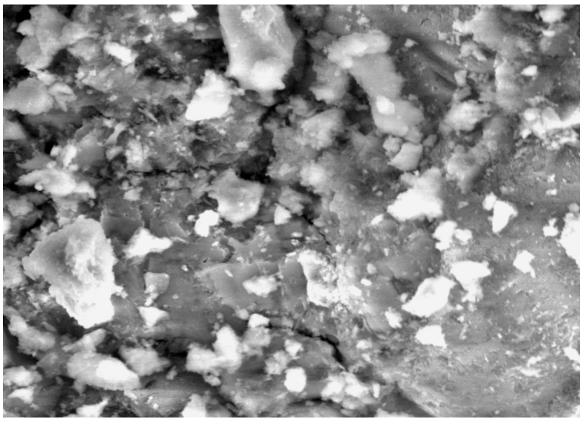

Figure 14: SEM Image of OPC replacement by EFA50%+10%NS with SPL at 28 days

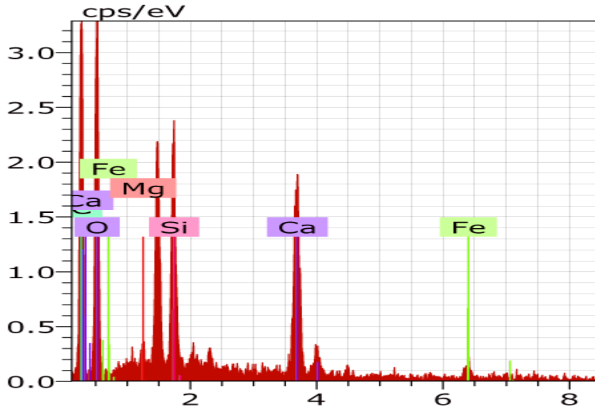

Figure 15: EDX analysis of EFA50%+10%NS with SPL at 28 days

5. Conclusions

The pivotal conclusion drawn from this study was 50% replacement of cement with Fly Ash reduces the strength and the durability properties of the mortar, but by the addition of Nano silica with the fly ash in 5% and 10%, these properties were significantly improved. The important conclusions drawn from this study were as follows:

- Replacement of cement with RFA and EFA reduced the workability of the mortar leading to increased Normal consistency of the mortar; this was due to the increase in reactive surface area in fly ash mortar and due to the angular particle shape of the fly ash mortar.
- The replacement with EFA is very efficient in improving the properties of the mortar than the replacement with RFA, and this is mainly because the EFA particles are finer than RFA particles. Thus it provides large reactive surface area than the RFA and the voids in the EFA mortar is lesser than voids in the RFA mortar.
- 50% replacement of cement with RFA and EFA reduced the compressive strength of cement mortar up to 42% and 10% respectively.
- Comparing to control mortar, early strength development in the fly ash mortar was low. Adoption of lower W/B ratio would enhance the mechanical and durability properties of the mortar.
- The capillary suction of water by the fly ash mortar is lesser than the control mortar. Control mortar showed poor acid resistance, while the fly ash mortar showed the better resistance to the acids.
- From SEM results it was concluded that the cement paste with EFA produces a denser structure than that of the paste with the RFA.

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