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Investigation on microstructural and compressive strength characteristics of a novel bio-admixture comprising Cassava Starch-Xanthan gum with cement mortar for building application

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ABSTRACT

Developing high-performing bio adhesives for enhancing the bonding strength of cement-based materials is highly desirable as they improve workability, corrosion resistance and durability of the concrete. The present effort aims at stemming a novel bio admixture comprising agricultural products Cassava starch and Xanthan gum as well examines the effect of incorporation of it into the cement. The composition of xanthan gum is varied in the starch mixture systematically in the ratios namely 1:0.25, 1:0.5 and 1:0.75. As-prepared bio admixtures are characterized through Fourier Transform Infra-Red spectroscopy, X-Ray Diffraction, Thermo Gravimetric Analysis, contact angle and Scanning Electron Microscopy-Energy Dispersive X-ray studies. These results demonstrated that presence of optimal amount of xanthan gum facilitates the binding nature of starch and promotes better bond strength as it significantly improved the bonding capacity as well as hydrophilic nature of starch thereby augmenting the bonding strength when used in cement mortar. The bio admixture filler material occupied the voids between the mortar matrix, thus reducing the porosity. The beneficial effect of as-prepared bio admixture has been reflected in the compressive strength that has been enhanced by 16.6% when optimized quantity (1.5 wt.%) of the bio admixture is added to the mortar.

Keywords: Biopolymers; Bio-Based Admixture; Starch; Xanthan Gum; Natural Adhesive; Improved Mechanical Strength.

1. INTRODUCTION

Admixtures are the essential component of concrete as they play a vital role towards altering interfacial properties and profoundly influence the workability, bonding strength, curing time of concrete [1, 2]. It is to be noted that achieving durability in concrete should be a very important consideration in the planning and building of new structures as well as in the assessment of the state of existing structures. It is well-documented in the literature that the way concrete interacts with its surroundings will affect the most likely causes of deterioration [3, 4]. Throughout the service life of the structure, concrete's resistance to chemical attack, abrasion, weathering, and other deterioration impacts is crucial, therefore concrete's endurance is significantly influenced by the materials used in construction. Green concrete preparation using bio-admixtures rather than conventional chemical admixtures has garnered a lot of interest.

Admixtures are added before or after the preparation of concrete, are of prime importance to fabricate desirable concrete design, chemical admixtures may be small organic compounds or chemical admixtures is where molecular structure adds the most value. Such admixtures give chemists the best opportunity to alter characteristics and increase performance through the targeted exploitation of structure/property correlations [5]. A diverse range of admixtures namely superplasticizer, accelerators/retardants, viscosity modifying agents air-entraining admixtures has been widely studied [6–8]. Although chemical admixtures influence the rheological and mechanical behavior of cement-based systems, they pose serious threat to the environmental pollution, which may have a negative impact on human health [9]. Superplasticizers, for instance, are mostly made of artificial, water-soluble polymers such as sulfonated naphthalene formaldehyde condensate, sulfonated melamine formaldehyde condensate, etc., that may cause toxic environmental issues. When inadvertent or on deliberately discharged into the environment, these non-renewable chemical admixtures that may have unfavorable environmental harmful impacts. For example, oil-based

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admixtures release formaldehyde that is harmful to the ecosystem. Certain chemicals like ammonia could be released in air from the concrete itself and chemical leaching from cementitious materials is harmful for aquatic life [10].

In the light of the abovementioned facts, recently the development of bio-based admixtures that may substitute chemicals and perform comparably has entered the picture with the growing interest in green concrete. Due to their widespread availability and straightforward manufacture, starches can be a practical option for bio-admixtures. Because they are renewable, sustainable green construction is guaranteed. Starch is the most abundant biopolymer that is widely explored as bio admixture for concrete due to ready availability, low-cost and eco-friendliness [11–14]. The sources of the starch in general are corn, cassava, sweet potato, sorghum potato, barley, rice, wheat, etc., chemical constitution and structure of starch depends on its source [15, 16]. The major components of starch are amylase and amylopectin, the proportion of these polymers differs depending upon the source. Further, starch was shown to be a set-retarding admixture for concrete that has heightened the compaction strength and workability of concrete [17]. Polymers which assist in the modification of rheological characteristics during the casting process are now essential components of several advanced concrete systems for that cement pastes with polysaccharides, acacia gum and cassava has been investigated [18]. Recently, an investigation was performed to determine to utilize rice husk ash in place of some of the cement and cassava starch (CS) as a natural additive in the creation of fresh and hardened concrete [19]. Researchers also investigated the effects of carrot extract on the properties of cement-based materials at both the macro and micro levels using a variety of detection techniques. Additionally, a novel and clean process for turning waste carrots into a novel natural additive for cement mortars was developed [20]. Besides, tannic acid has been explored as a molecular-grade admixture to modify mortar and reduce the total porosity [21]. The mechanical properties of the hardened concrete such as compression is improved by adding bamboo ash with conventional concrete mix [22]. A natural biopolymer chitosan was incorporated in different dosages with the cement. The results of the study indicated that chitosan can be considered to be used a bio admixture [23]. Since biopolymer are easily extractable or can be synthesized in a single step or dual step processes through the laboratories and are highly cost effective, the application of these biopolymers as an admixture is promising.

The proposed work is preliminary research on preparation, characterization, and application of locally availed Cassava starch-xanthan gum admixture with enhanced adhesive characteristics. Xanthan gum is a biodegradable negatively charged hydrophilic polymer formed as a fermented product of gram-negative bacterium Xanthomonas campestris. Even though these cross-linked biopolymeric architectures bring about improved thermomechanical properties, the use of starch-xanthan gum is explored in the diverse fields such as food science, drug delivery etc., Xanthan gum is an excellent hydrocolloid for stabilizing aqueous dispersions, emulsions, and suspensions that exhibit highly pseudoplastic flow behavior with has good bio adhesive properties. The anionic character of it arises due to the existence of both glucuronic acid and pyruvic acid groups in the side chain. The research world of civil engineering has developed various new alternative materials as either an additional material or a substitute material to the conventionally available admixtures [16]. Also, another set of researchers are attempting the usage of chemical and mineral admixtures to improvise the properties of the cement, modify the viscosity of concrete. Mostly these materials, help in a formation of dense C-S-H gel, acts as a filler material thus reducing the porosity of the concrete medium and thus increasing the strength of the mixture. With the intention of improving sustainability by employing naturally renewable resources, the present research assessed the potential use of Cassava Starch-Xanthan gum as an addition in cement concrete systems. It significantly affects the cement's workability, durability, and setting time and in contrast to chemical admixtures, it has no negative environmental effects.

2. MATERIALS AND METHODS

2.1. Bio-admixture preparation

Our geographical location is one of the largest producers of cassava globally. Locally available powdered tapioca starch was directly procured from sago industries, Salem. The xanthan gum was procured from nature's velvet brand, the purity of xanthan gum was about 99%. Doubly distilled water is used for all the experiments 4 wt.% of Cassava starch is dissolved in the water under continuous stirring at the rate of 600 rpm for 4h. Pre-determined amount of xanthan gum is added dropwise into the Starch and allowed to stir for 2h. Xanthan gum is incorporated into Cassava starch and its composition varied as 1:0, 1:0.25, 1:0.5 and 1:0.75. As-prepared starch-xanthan gum samples comprising 1:0.25, 1:0.5 and 1:0.75 ratio was designated as pure starch (PS), SX1, SX2 and SX3 respectively. The samples were filtered, dried in a hot-air oven at 70 °C for 4 hours, ground using a mortar and pestle finely and used for further studies. The physico-chemical properties of as-prepared samples were performed using Fourier transform infrared spectroscopy (FTIR), X-Ray diffractogram (XRD), Thermogravimetric analysis (TGA), Contact angle measurements and Scanning electron microscopy (SEM), Elemental analysis (EDS) characterization. Pure starch (PS) is used for comparison studies. Table 1 depicts proportion of mixing biopolymer components. By using KBr pellets, the FT-IR Shimadzu 8400S spectrophotometer measured the infrared spectra between 4000 and 400 cm^{-1}. TGA scans were carried out from 25 °C to 1200 °C under a heating rate of 2 °C/min under nitrogen inert atmosphere. The instrument model used for the study was NETZSCH STA 449F3. The produced bio admixture is evaluated using an ethanolic solution and a scanning electron microscope (SEM)-EDAX. An X-Ray Diffractometer (Model Philips Pan Analytical) was used to measure XRD pattern samples using powder X-ray Diffraction (XRD) patterns with the wavelength of 1.5406 for CuK radiation. To ensure that these images were accurate depictions of the surface morphology, many photographs were taken at varied locations and scan ranges.

2.2. Compressive strength measurements

An experimental study was carried out by using the bio-admixture composition as an addictive in the cement mortar paste. The as-prepared bio-admixtures namely SX1, SX2 and SX3 in the varied proportion of 0.5 wt.%, 1 wt.%, 1.5 wt.% and 2 wt.% by the weight of the cement is added into Portland pozzolanic cement (PPC) confining to IS 1489:1991 was used along with fine aggregate confining to IS 383: 1970 was used. The particle size distribution of the M sand was within the zone II. The elemental composition of the Portland pozzolanic cement is shown in Figure 1.

The procedure to conduct the following test was as per IS 4031 (Part 6): 1988. To compare the results, compressive strength studies are performed with the addition of pure starch. One part of cement was mixed with three parts of fine aggregate to obtain the cement paste. The materials were hand-mixed in a dry state for 15 min. The mixing was carried out continuously until the sample attained a homogenous texture. The temperature of all the dry ingredients and the environment conditions was maintained at 27 ± 2 °C. The relative humidity was also maintained at 65 ± 5% in the room. The pastes were added into a cube mould of size 70.6*70.6*70.6 mm^3 Figure 2.

Table 1: Bio-admixture ratio.

SAMPLE	STARCH CONTENT(g)	XANTHAN GUM (g)	MIX RATIO
Pure starch	2	0	1:0
SX1	2	0.5	1:0.25
SX2	2	1	1:0.5
SX3	2	1.5	1:0.75

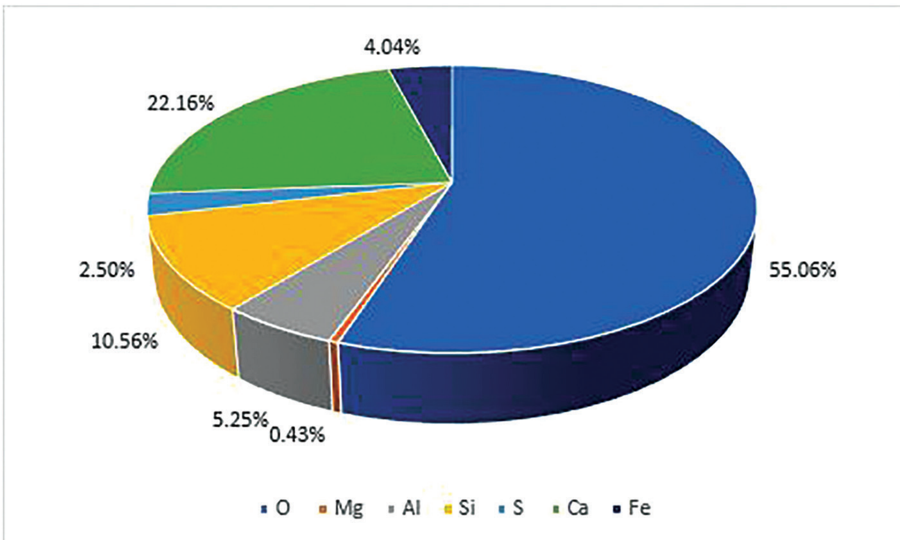

Figure 1: Elemental composition of PPC.

Figure 2: Casted specimens.

Table 2: Compression strength test results.

SL. NO	SPECIMEN ID	COMPRESSIVE STRENGTH (MPa)	
		7th day	28th day
1	Control specimen	22.78	32.40
2	0.5% – PS	20.45	29.63
3	0.5% – SX1	15.37	28.04
4	0.5% – SX2	17.52	30.09
5	0.5% – SX3	20.51	33.51
6	1% – PS	20.20	28.18
7	1% – SX1	16.49	27.30
8	1% – SX2	19.82	31.12
9	1% – SX3	22.94	34.41
10	1.5% – PS	20.70	29.30
11	1.5% – SX1	17.39	27.60
12	1.5% – SX2	20.78	33.73
13	1.5% – SX3	22.50	37.82
14	2% – PS	19.82	28.49
15	2% – SX1	16.79	26.66
16	2% – SX2	20.04	33.07
17	2% – SX3	21.45	35.97

The behavior of the cement mortar cubes under compressive loading were studied on the 7th day and 28th day. The cured specimens were placed in compression testing machine without any packing on the sides and the load was applied steadily and uniformly at a rate of 35 $N/mm^2/min$ as shown in Figure 3.

The compression strength of each composition was determined from the average of three sample on the age of curing. The results are mentioned in the Table 2.

3. RESULTS AND DISCUSSION

The results of the studies conducted are interpreted and these studies reveal suitable composition of biopolymeric mixture that could be used as bio admixture.

Figure 3: Specimen under loading.

3.1. FTIR studies

Figure 4 represents FTIR spectra of PS, SX1, SX2 and SX3 samples. A set of characteristics set of peaks appearing at 3700 cm^{-1}, 2960 cm^{-1}, 1710 cm^{-1} and 1515 cm^{-1} are due to different functional groups present in the starch namely O-H stretching vibration, C-H stretching of CH_2, O-H bending vibration and CH_2 in-plane bending vibration respectively [24]. The stretching C-O vibrations at 1020 cm^{-1} and the stretching C-H vibrations from CH were among the starch's distinctive peaks group at 2960 cm^{-1}, and O–H stretching vibrations are responsible for the peak at 3700 cm^{-1}. A further peak, located at 1648 cm^{-1}, was indicative of starch and resulted from the stretching of C-O-C vibrations and the first harmonic OH bending. It is apparent from SX1, SX2 and SX3 spectra that the spectral features are nearly similar but different from that of PS. After incorporating xanthan gum, few spectral bands have been modified and rest of the remain unaltered. Stretching of the C-H molecule is characterized by the band at 2928–2932 cm^{-1}. The vibrational stretching associated with free, inter, and intra molecule bound hydroxyl groups is responsible for the appearance of a wideband owing to hydrogen linked hydroxyl group (O-H) at 3420–3434 cm^{-1} is appeared in all the spectra. The bound water found in the starch and xanthan gum is what causes the peaks at 1645 cm^{-1}. After the addition of xanthan to the starch in the ratio of 1:0.25 a shift in the peaks of the spectroscopy is found.

3.2. TGA

The thermal stability of bio admixture has been compared with pure starch as shown in Figure 5. During the entire heating operation, weight-loss of the material was investigated and recorded at regular time intervals. The degradation takes place in three different phases, removal of moisture, loss of volatile matter and loss of fixed carbon. The results have been plotted for Mass vs Temperature shown. The mass of the sample is represented in '%' along Y axis and the temperature is represented in '°C' along X axis. At initial phase (around 100 °C), the water molecules and volatile matter are lost form the material, quantitative degradation is less for PS as compared to other samples under study, indicating more hydrophilic character of starch-xanthan gum composite [25]. At 100 °C, weight-loss associated with PS, SX1, SX2 and SX3 is 5.67%, 10.11%, 10.88% and 11.24% respectively. Usually, the cleavage of the bond takes place in the second stage, the loss of mass of the pure starch, SX1, SX2 and SX3 at the second stage are 64.47%, 55.27%, 52.06% and 51.62% respectively. Xanthan–starch hybrid polymer backbone exhibited the decomposition process at higher temperature in relation to pure starch. Followed by it, the final phase of degradation is due to the structural collapse of polymers and carbonization of organic matters and non-volatile constituents. The remnant weight-loss is noted as 10.74% for PS which is lower than that of SX1 (18.23%), SX2 (27.36%) and SX3 (20.79%) samples. These results clearly proved that thermal stability of pure starch has been enhanced after incorporating xanthan gum due to string

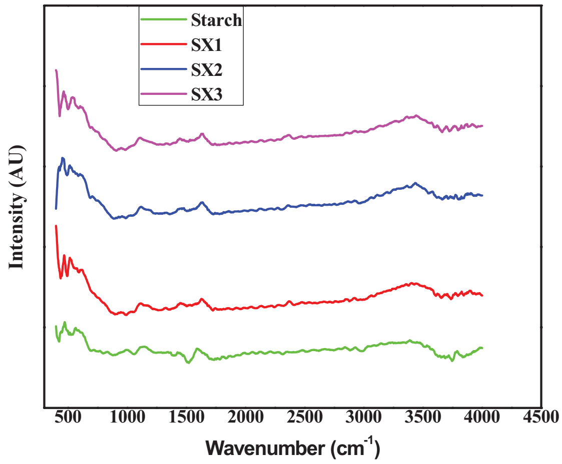

Figure 4: FTIR spectra of PS, SX1, SX2 and SX3 samples.

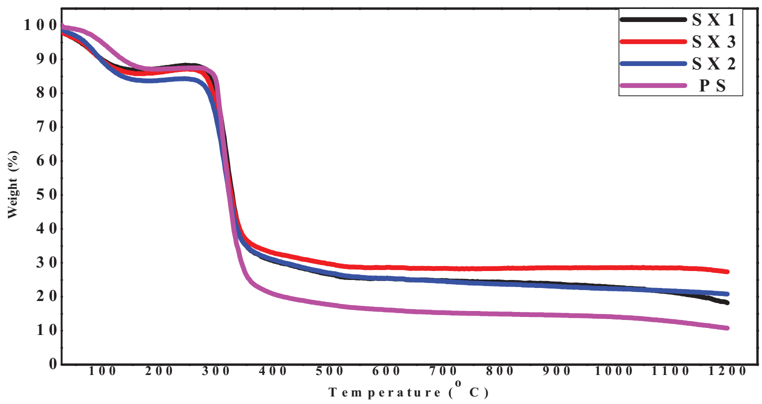

Figure 5: TGA curve of PS, SX1, SX2 and SX3 samples.

interaction among constituting polymers. Since the extent of thermal degradation is less for SX samples, it could be deduced that introduction of xanthan gum into the starch improve thermal stability. The existence of favorable intramolecular interaction between starch and xanthan gum through hydrogen bonds, Vander Waals force and electrostatic interactions might have positively contributed for the improved thermal stability.

3.3. XRD

XRD pattern of PS, SX1, SX2 and SX3 are as shown in the Figure 6 that reveals modified crystalline packing of pure starch as compared to that of starch-xanthan composite. XRD form of starch displayed a sharp peak at 21.6° with a strong diffraction signal, and four other peaks around the area 15°, 22°, 23° and 24° appearing both at crystalline and amorphous regions representing its semi-crystalline nature and typical C-type crystallinity pattern as reported earlier. Based on its main constituents, amylose and amylopectin, starch is a semicrystalline substance that contains both the crystalline and amorphous phases. The crystalline portion of starch is made by the linear structure of amylose, while the amorphous form of starch is generated by the branching structure of amylopectin [26, 27]. When xanthan gum is incorporated into starch matrix, crystallinity has been decreased to a certain extent because of amorphous nature of xanthan gum, however the major peaks remained unaltered. The SX1, SX2 and SX3 samples exhibited reflections at 15.3°, 21.9°, 23.8° and 24.3° which are slightly deviated from that of PS. It is inferable from the results that xanthan gum has been interacted with starch and modified packing pattern of polymer chains and resulted in variation of crystalline packing within the polymer matrix. It is notable that the observations are in accordance with the earlier reports [28, 29].

3.4. Contact angle studies

Contact angle (θ) theta is a quantitative measure of wetting of solids by a liquid. The contact angle is geometrically defined as the angle formed by the liquid at the three-phase boundary, where a liquid, gas and solid

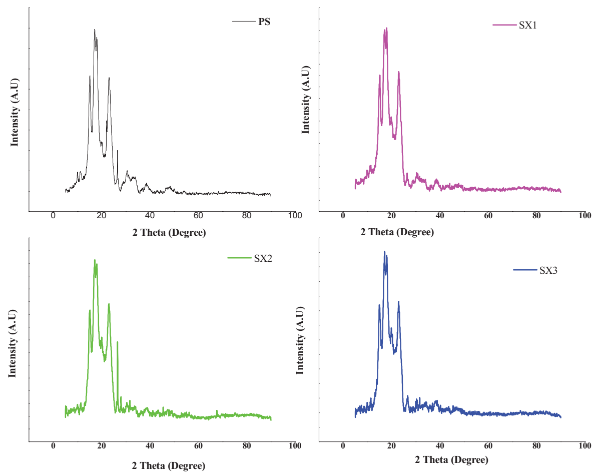

Figure 6: XRD pattern of PS, SX1, SX2 and SX3 samples.

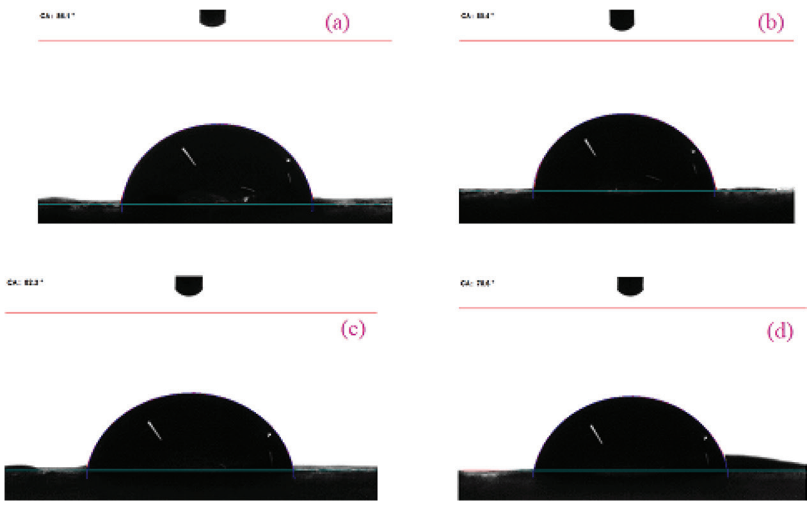

Figure 7: The contact angle of: (a) PS, (b) SX1, (c) SX2 and (d) SX3 samples.

intersects [30, 31]. Water contact angle will immediately give an indication of the wettability of the solid. If the measured contact angle is above 90 degrees, the solid is said to have poor wetting and is termed as hydrophobic. If the contact angles are below 90 degrees, the material is stated as hydrophilic. The contact angle of PS, SX1, SX2 and SX3 determined to be less than 90 degree which represents the hydrophilic nature of the material as represented in Figure 7a-d which designates adhesive nature of all samples under study. As evident from the figures, after introducing xanthan gum, hydrophilic nature of starch gets improved the value of contact angle has been decreased with the increase in concentration. After mixing with xanthan gum, contact angle has been decreased that has high absorption nature which directly indicates that the sample has good binding nature. As per the results, the SX3 composition has lesser contact angle than the SX1 and SX2 composition, which proves that this sample has better bonding nature and will be suitable for bio admixture application.

3.5. SEM – EDX studies

From the above-mentioned study results SX3 composition proved to have a better binding nature than the others. So, the SEM and EDX studies were carried out on this sample along with the pure starch sample for comparison. SEM images under different magnification of PS and SX3 have been depicted in Figures 8 and 9 respectively that provide insight into of internal structure to further recognize the morphological change.

As visible from the images, both PS and SX3 depict granular morphology with either smooth or low-roughened surface. On the starch granules, it is confirmed that there are rougher zones and relatively smooth sections with low surface roughness. Several pin holes could be seen on the surfaces of the starch granules that amylase was attacking. The shape of these granules is either oval, spherical or irregular polygonal in form,

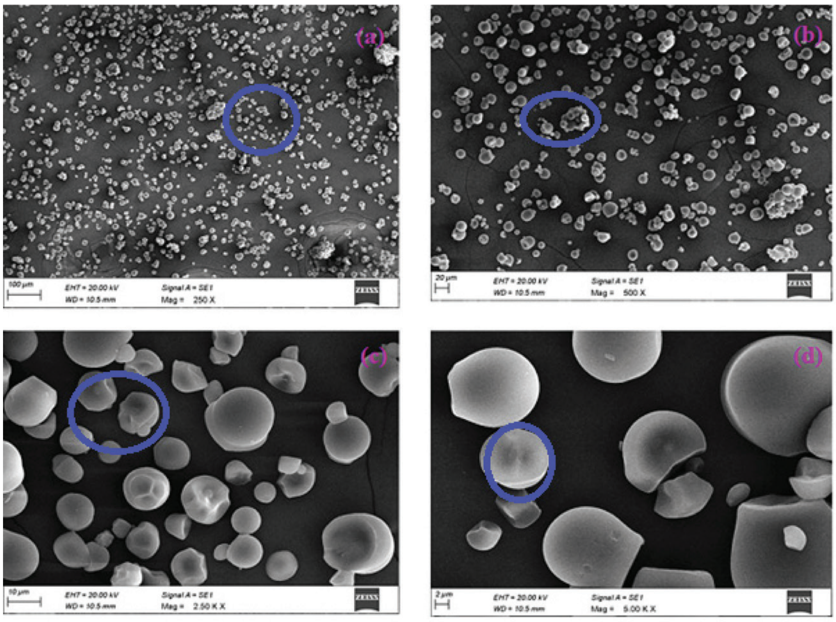

Figure 8: The SEM images of PS under different magnifications.

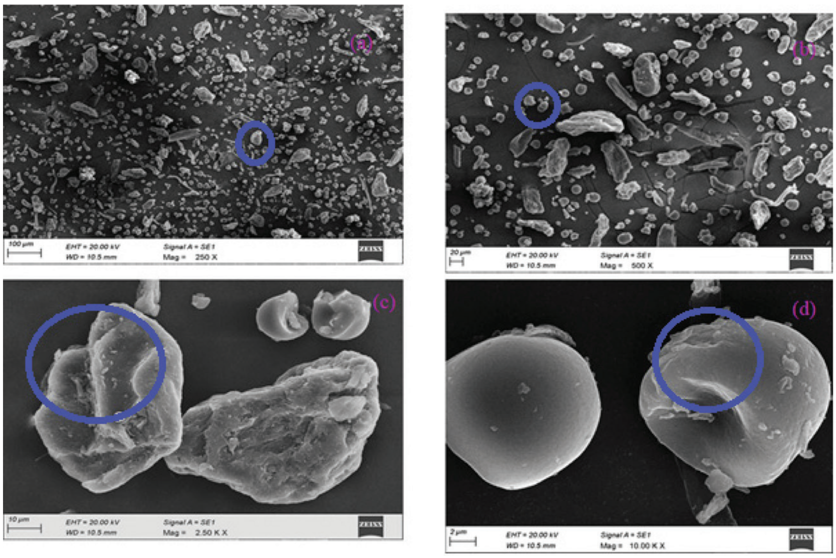

Figure 9: The SEM images of SX3 under different magnifications.

therefore both PS and SX3 present similar morphology. However, the average granular size is higher for SX3 is 10.5 µm which is only 4.5 µm for PS. It is noteworthy that the starch granules were well-defined and do not undergo any structural damage due to incorporation of xanthan gum. The salient features associated with starch and xanthan gum modified starch are being highlighted in the SEM images. These images further confirmed the formation of mixed polymeric network. From the EDX results, the chemical composition of SX3 composition is shown in Figure 10. The figure shows the presence of carbon, oxygen, alumina, calcium, silica, iron, and some other elements.

3.6. Compression strength studies

Figure 11 represents the compression strength results of cement paste comprising pure starch and bio admixtures of different composition. On the interpretation of the results, the addition of admixture improves the compressive stress of the cement mortar cube. The sample of 1.5 wt.% addition of bio-admixture with the starch and xanthan gum ratio of 1:0.75 (SX3) composition performed better in the 7th and 28th day compression test.

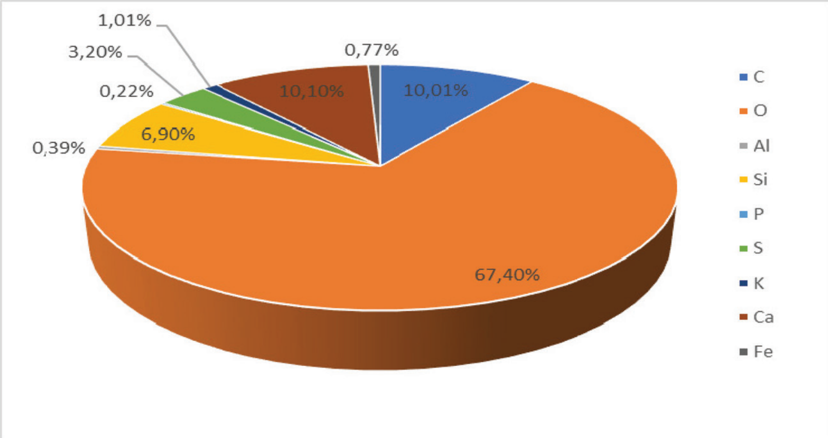

Figure 10: Elemental composition of SX3 admixture.

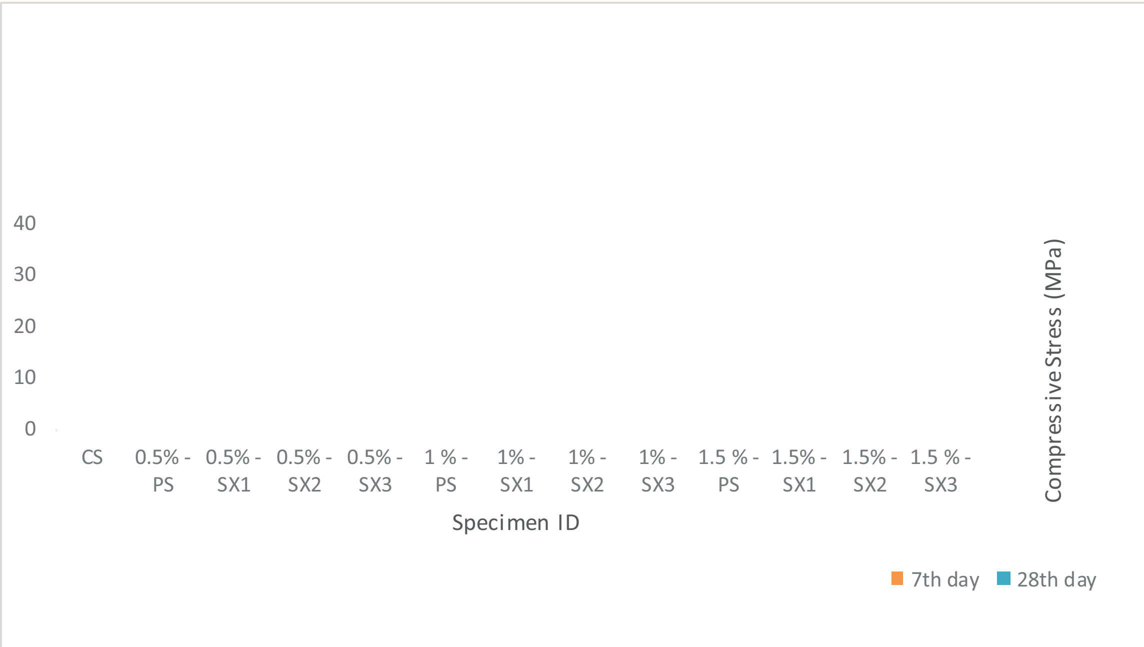

Figure 11: Compression strength test data.

The compression strength of SX3 has been increased by 5.5 MPa at 28th day of curing. The increase in compressive strength up to the 7th day was not much significant. The results state that addition of bio-admixture does not contribute to early strength. The results of the specimens with SX3 ratio of bio-admixtures, namely 2 wt.% - SX3, 1 wt.% - SX3, and 0.5 wt.% - SX3 had developed in withstanding the compressive load by 3.5 MPa, 2 MPa and 1.1 MPa, respectively. The specimens after failure are shown in Figure 12.

The quantity of xanthan gum with the starch directly affects the outcome. It is to be noted that as the quantity of xanthan gum is increased there is steady increase in the compressive strength of cement. The starch and the xanthan are highly crosslinked in the composition of higher ratio mix of starch and xanthan (1:0.75). The chemical composition of the biomaterials contributes to the formation of a sturdy covalent bonding. The molecular weight and the length of the polymer chain is also amplified. These properties appropriately enhance the density of the bio-admixtures matrix. As a result, the force required to remove the bonding is more, hence this composition can withstand more load. Consequently, there is an increase in the compression strength of the cement mortar. The similar results are observed.

Figure 12: Specimens after loading.

The percentage of addition of the bio-admixture was also examined in this work, 1.5 wt.% addition of bio-admixture by the weight of the cement content proved to be optimum compared to that of 0.5 wt.%, 1 wt.%, 2 wt.%. It is inferable form the data that compression strength keeps increasing upon increasing the wt.% of bio admixture up to 1.5 wt.% but it starts declining when increased to 2 wt.%. The highly effective adhesiveness of the bio-admixture creates a strong cohesiveness in between the particles of the mortar matrix. The biopolymer reacts with the water and generates a thick amorphous gel. The polymeric gel occupies the voids between the cement and fine aggregate of the mortar mix. The presence of silica is as shown in the Figure 10 contributes to formation of a highly dense calcium silicate hydrate gel and improves the packing density of the cement paste. Consequently, the highly dense C-S-H medium exhibits fewer voids thus facilitating a strong binding among the materials. Since the porosity of the mixture is decreased, this specimen possesses higher load-bearing capacity than the control mix specimen comprising a greater number of pores in its structure.

4. CONCLUSION

The work has provided a new insight into the connection between molecular structure or chemical composition of admixtures and the macro performance of concrete. It is a well-known fact that the bond strength of the cement is the most important characteristic in determining the serviceability of any type of concrete structures. The physico-chemical characterization studies and real time analysis proved that inclusion of xanthan gum into starch has markedly enhanced the bonding strength of the bio admixture. From the above discussions, among the different proportions of the biopolymer mix, the bond strength of the composition with starch and xanthan in the ratio of 1:0.75 (SX3) composition proved to be promising. This mix can be used with various grades of concrete, or cement paste. The biopolymer acts as a natural admixture to enhance the mechanical strength of the cement-based product. The conclusion was made from the following considerations

- The FTIR spectroscopy has a distribution of peaks in the singe bonded region, double bonded region, and the triple bonded region and the intensities of the peaks are also higher comparatively.
- The SX3 composition has lesser contact angle than the SX1 and SX2 composition, which proves that SX3 sample has better bonding nature and will be suitable for the proposed research work.
- A lower percentage of cleavage has occurred in the SX3 sample, indicating that the mix has lost its bonding nature in a fewer amount that the others even at high temperatures. This makes the SX3 mix better binding mixtures.
- A better compression strength at 28^{th} days of curing was acquired for cement mortar with the 1.5 wt.% of addition by the weight of the cement and 1:0.75 ratio of starch: xanthan gum. This composition is determined as the optimized dosage.

5. ACKNOWLEDGMENTS

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