The application of Natural Organic Additives in Concrete: Opuntia ficus-indica.

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Abstract. The use of synthetic additives as setting retarders and plasticisers has over the past years become a standard practice in mortar mixtures, despite their potential adverse environmental effects. Since antiquity, man has used organic additives ranging from plant and animal-derived extracts, to improve the setting properties of mortars. These practices are slowly being revived and investigated scientifically to promote the use of materials with lower impacts on the environment. Amongst the many different types of plant-derived extracts that have been used as mortar and concrete additives, the prickly pear (*Opuntia ficus-indica*) mucilage extract, is popular in Meso- and South-America. The scientific basis of these additives lies in the hydrating properties of the mucilage polysaccharide complex. The purpose of this research was to prepare *Opuntia ficus-indica* (OFI) extracts in different forms and incorporate them in cement pastes and mortar mixtures by either replacing the water in the mixture with OFI mucilage or by replacing the cement in the mixture with OFI lyophilised powder. The inclusion of *Opuntia ficus-indica* additives in cement-based mortars increased their performance in terms of strength for both water and powder replacements. Conversely results in cement pastes showed that *Opuntia ficus-indica* additives weakened the cement paste samples in terms of strength in both water and power replacements. Results for both cement-based mortars and cement pastes showed an increase in setting time for both water and powder replacements, indicating that *Opuntia ficus-indica* additives could be potentially used as retarding agents.

Keywords: Natural organic additives, Opuntia ficsu-indica, setting time, retarder

1. Introduction

Concrete structures are expected to resist weathering action, chemical attacks, and physical action while still maintaining their desired engineering properties over their expected lifetime. The development of micro-cracks and subsequent penetration of fluids into these structures often leads to degradation and failure before the expected life-time. Durability performance is in part related to the setting and curing properties of the concrete, cement paste or mortar at the time of manufacture. Rapid setting of the cement affects the micro-structure of these materials.

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The setting properties of mortars and concrete can be controlled using admixtures including different types of organic extracts. Retarding admixtures as the name implies help to retard the setting of the cement especially in the case of high ambient working temperatures associated with hot weather concreting. By using retarders, the heat generated from the exothermic hydration reactions is allowed to dissipate and the desired crystalline structure can develop.

This retarding action is usually provided by the addition of carbohydrates including cane sugar, as well as other organics and soluble zinc salts amongst many others. The dosage of any retarding admixture is determined through trial mixes. The effect of these admixtures depends on the chemical composition of the cement and the admixture performance. For example, a very low dosage of cane sugar (0.05 %) can delay setting time by as much as 4 hours. However, a larger quantity can completely prevent the cement from setting. It is also interesting to note that research has shown that some admixtures can act as retarding agents at low percentages but can also act as accelerators if higher percentages are used (Neville and Brooks 1987). Though the mechanism of retardation is not fully understood, it is known that admixtures modify crystal growth. The inclusion of these retarding admixtures influences greatly the rate of strength gain of concrete, and there is some evidence that the final strength may be improved. Several researchers have observed a higher ultimate strength of concrete when the rate of strength gain at the early stages is retarded (Chandra et al 1998). Furthermore, if the retarding admixture is also a water-reducing admixture, a higher strength can be achieved by decreasing the water to cement (W/C) ratio (Neville and Brooks 1987).

Despite their potential adverse environmental effects, the use of synthetic additives as setting retarders and plasticisers has over the past years become a standard practice in mortar mixtures. Organic additives have been used since ancient times to improve the working properties and durability of lime-based mortars and concrete. These practices are slowly being revived and investigated scientifically to promote the use of materials with lower impacts on the environment. Amongst the many different types of plant-derived extracts that have been used as mortar and concrete additives, the prickly pear (*Opuntia ficus-indica*) mucilage extract, is popular in Meso- and South-America. Previous studies have identified *Opuntia ficus-indica* (OFI) extract as a potential concrete and mortar additive. It is an ideal organic additive as the plant is widely available in warm climates and it can be easily harvested and processed to yield the additive. The scientific basis of these additives lies in the hydrating properties of the mucilage polysaccharide complex.

Chandra et al., (1998) studied the potential benefits of cactus extracts in mortars. They used X-ray diffraction and the scanning electron microscopy to understand the hydration process of the mortar with mucilage addition. Moreover, they also studied flexural strength, compressive strength and water absorption with regards to OFI addition. From the diffractograms, it was noted that the calcium hydroxide peaks decrease with the addition of the OFI extract (Chandra et al 1998). The conclusion was that the reduction in peaks was an indication that the calcium hydroxide could be reacting with components of the OFI extract forming calcium-based organic complexes. Chandra et al (1998) have shown that the natural OFI polymer influences the hydration process of tri-calcium silicate and hinders the formation of big crystals of calcium hydroxide. This research thus confirmed that the influence of the mucilage on the hydration process is not an artifact.

Martínez-Molina, Torres-Acosta, Celis-Mendoza, & Alonso-Guzman (2015) investigated the physical properties of cement-based mortars containing OFI extracts. In this case, a dehydrated powder was used as a replacement for the cement. This research was conducted over a period of 900 days focusing on four types of tests, total void content, compressive strength, ultrasound wave propagation and wet electrical resistivity. The authors concluded that the mortar fluidity was observed to decrease with the increase of the organic extracts and therefore more water was needed to obtain mortars with the same workability. Nevertheless, the hardened properties of the mortars with dehydrated OFI showed a marginal improvement in hardened properties even at low percentage replacements.

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Several techniques are recorded in the literature to obtain the mucilage from the *Opuntia* pads (cladodes) which are modified stems that serve as a water storage and the main photosynthetic organ in the absence of true leaves. Each method of OFI extract production has its own set of constraints varying from low to the high energy input and this is in turn related to the amount of residual water in the final product.

The purpose of this research was to produce *Opuntia ficus-indica* (OFI) extract by two different techniques and incorporate them in cement pastes and mortar mixtures by either replacing the water in the mixture with fresh OFI mucilage, or by replacing the cement in the mixture with OFI lyophilised powder.

2. Materials and Methods

2.1. Opuntia ficus-indica Additions

Water and powder replacement methods were investigated. For both methods, the inner white section of the *Opuntia ficus-indica* pads or cladodes was used to produce the mucilage. These cladodes have an estimated water content of 90%. For the water replacement, the inner part of the cladode was finely diced, weighed and boiled. The resulting mucilage was then sieved to remove the large fibers, and was refrigerated until used in the cement pastes and mortar mixes. For the powder replacements, a freeze-drying process was used on the inner white section of the pads, followed by crushing of the material using a mortar and pestle. The weight of powdered mucilage was recorded before and after sieving through a 150 μm mesh. The process of lyophilsation is illustrated in Figure 1.

Figure 1. Opuntia ficus-indica lyophilising process: a) Opuntia ficus-indica plant; b) inner white section of the cladode; c)inner part of the cladode subject to the freeze-drying process; d) Opuntia

2.2. Paste and Mortar Specimen Preparation

2.2.1. Paste Sample Preparation

A total of sixty 20 x 20 x 20 mm paste cubes, with a water to powder (W/P) ratio of 0.28 and 0.35 were prepared in accordance with EN 196-1:2016. The consistency of the control sample was obtained through a number of trial mixtures until a consistency of (6 ± 2) mm was obtained. The same mixture was used for the different percentage replacements. In the case of the cement pastes containing powder replacements, the mixture was not workable for the same W/P Ratio. Therefore, a higher W/P Ratio was used for these samples. Once cast, the samples were demolded after 24 hours and testing carried out after 3, 7, 28 and 90 days from casting. In the case of the power replacements, the samples were demolded after 48 hours, due to a prolonged retardation of setting time. Once demolded, samples were placed in water at a temperature of ±20°C until testing. Tables 1 and 2 give details of the composition of the cement pastes.

Table 1. Mixture proportions for cement pastes containing OFI Water Replacements at 0.28 W/P Ratio

Type	OFI Percentage (%)	OFI Content(g)	Mucilage(g)	Water (g)	Cement (g)	W/P Ratio
Liquid [a]	0		0	140.00	500	0.28
Liquid [a]	2.5	3.34	3.45	136.51	500	0.28
Liquid [a]	5	3.34	7.00	133.06	900	0.28
Liquid [a]	7.5	3.34	10.50	129.56	900	0.28
Liquid [a]	10	3.34	14.00	126.82	900	0.28
Liquid [a]	20	3.34	28.00	112.07	900	0.28
Liquid [a]	40	3.34	56.00	84.02	900	0.28
Liquid [a]	60	3.34	84.00	54.04	900	0.28

[a] Boiled + Liquidised

Table 2. Mixture proportions for cement pastes containing OFI Powder Replacements at 0.35 W/P Ratio

Type	OFI Percentage (%)	Powder(g)	Water(g)	Cement(g)	W/P Ratio	W/C Ratio
Powder [a]	0	0.0	175	500	0.35	0.350
Powder [a]	0.5	2.5	175	497.5.	0.35	0.352
Powder [a]	1	5.0	175	495	0.35	0.354
Powder [a]	1.5	7.5	175	492.5	0.35	0.355
Powder [a]	2	10	175	490	0.35	0.357

[a] Lyophilised Powder

2.2.2. Mortar Prism Sample Preparation

Mortar prisms were prepared, in accordance to EN 196-1:2016. Each mixture was used to cast 72, 40 x 40 x 160 mm prisms to be used for the determination of the mechanical properties including the ultrasonic pulse velocity, flexural strength and compressive strength of the mortars. Tables 3 and 4 give details of the composition of the mortar mixtures. The mortar prisms were produced using Portland cement (Type CEM 1, 42.5 R) and a Coralline Limestone fine aggregate (4 mm maximum nominal size), with a W/P ratio of 0.7.

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Table 3. Mixture proportions for mortar samples containing OFI Water Replacements at 0.7 W/P Ratio

Type	OFI Percentage (%)	OFI Content(g)	Mucilage(g)	Water (g)	Cement (g)	Sand(g)	W/P Ratio
Liquid [a]	0	0	0	630	900	2700	0.7
Liquid [a]	2.5	3.34	15.75	614.25	900	2700	0.7
Liquid [a]	5	3.34	31.5	598.5	900	2700	0.7
Liquid [a]	10	3.34	63	567	900	2700	0.7
Liquid [a]	20	3.34	126	504	900	2700	0.7
Liquid [a]	40	3.34	252	378	900	2700	0.7
Liquid [a]	60	3.34	378	252	900	2700	0.7

[a] Boiled + Liquidised

Table 4. Mixture proportions for mortar samples containing OFI Powder Replacements at 0.7 W/P Ratio

Type	OFI Percentage (%)	Powder(g)	Water(g)	Cement(g)	Sand(g)	W/C Ratio	W/P Ratio
Powder [a]	0	0.0	630	900	2700	0.7	0.0
Powder [a]	0.5	4.5	630	895.5.	2700	0.7	4.5
Powder [a]	1	9.0	630	891	2700	0.7	9.0
Powder [a]	1.5	13.5	630	886.5	2700	0.7	13.5
Powder [a]	2	18.0	630	882	2700	0.7	18.0

[a] Lyophilised Powder

3. Results and Discussion

3.1. Fresh and Hardened Properties of Cement Pastes

Results indicate that all cement pastes samples containing both liquid and powder OFI replacements, exhibited a general decrease in flow, which effect was exacerbated with increased percentage replacement. With a decrease in flow, an increase in consistency was also noted for all the percentage replacements. Results are illustrated in Figures 2. This increase in cement paste consistency may be attributed to the presence of polysaccharides in OFI (Ribeiro et al. 2010). This follows because, the type of branching polysaccharides present in OFI gives rise to a matrix structure with high water-retaining characteristics which indeed is referred to as mucilage. Therefore, and it is safe to assume that some of the water present in the mixture finds itself in the polysaccharide matrix thus reducing flow and increasing consistency.

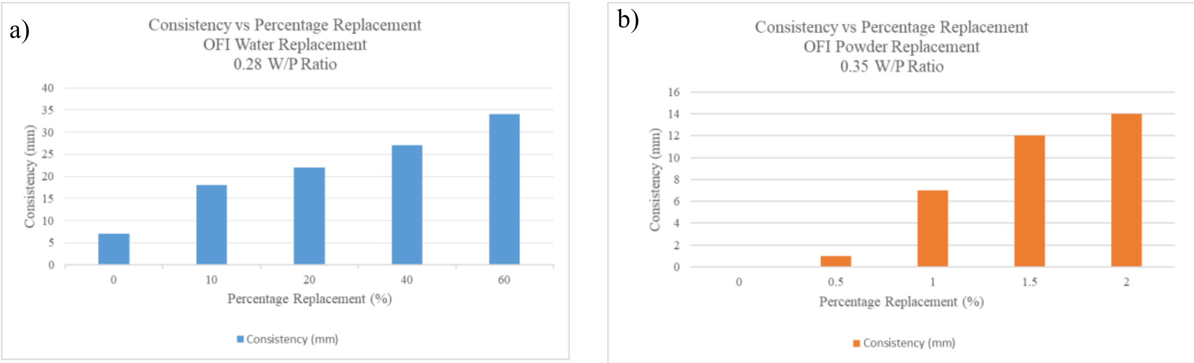

Figure 2. Graphs showing results for cement pastes consistency vs OFI percentage replacement for a) OFI water replacement with a 0.28W/P Ratio and b) OFI Lyophilised Powder Replacement with a

Graphs in figure 3 refer to the setting times for pastes containing OFI additions. As can be observed, all samples containing OFI additions exhibited an increase in setting time. In the case of OFI water replacements, the low percentages led to values close to the control sample. The largest increase was observed for 40% and 60% water replacement with an increase of 31% and 43% respectively. In the case of the cement pastes containing OFI powder replacement, the increase in setting time was considerably higher. The greatest increase was observed for 1% percentage replacement with a 75% increase for both the initial and final setting time. However, even though the results show that the highest increase in setting time was observed at the 1% percentage replacement, visual observation showed that the higher percentages (1.5%- 2%) took longer to consolidate. Indeed, these samples were still fragile when removed from the mould after 2 days of curing, when compared to the other percentage replacements.

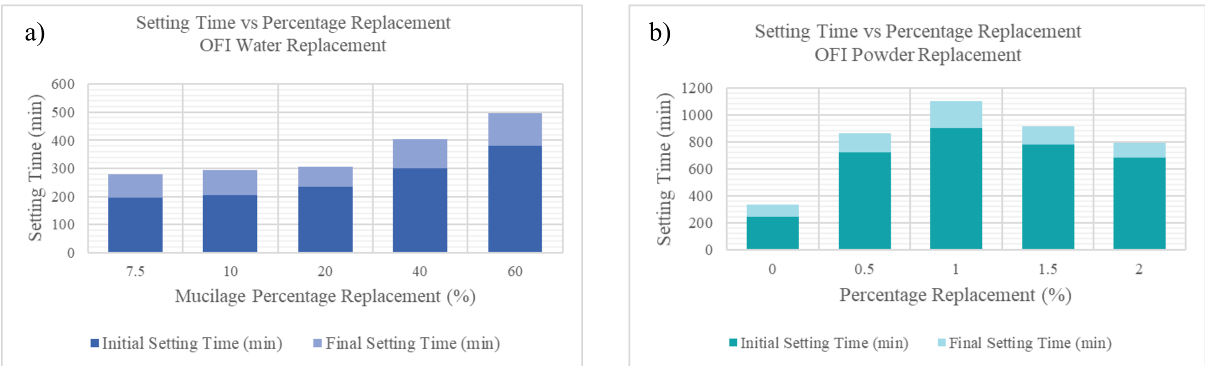

Figure 3. Graphs showing results for cement pastes setting time vs OFI percentage replacement for a) OFI water replacement with a 0.28W/P Ratio and b) OFI Lyophilised Powder Replacement with a

This increase in setting time, led to a delay in strength development at early age, with all cement samples containing OFI additions showing a decrease in strength with respect to the control sample also monitored at an early age. With time, the strength of OFI samples also increased, but it did not surpass the strength of the control sample. Moreover, at 3 and 7 days, the samples containing higher OFI percentages failed and crushed immediately when subjected to compression. This is illustrated in Figure 4 where compressive strength over time is compared.

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This increase in setting time shows the potential advantage of using OFI as a retarding agent when casting in high ambient temperatures. Concreting in hot weather presents various challenges and retarding agents reduce the rate of hydration.

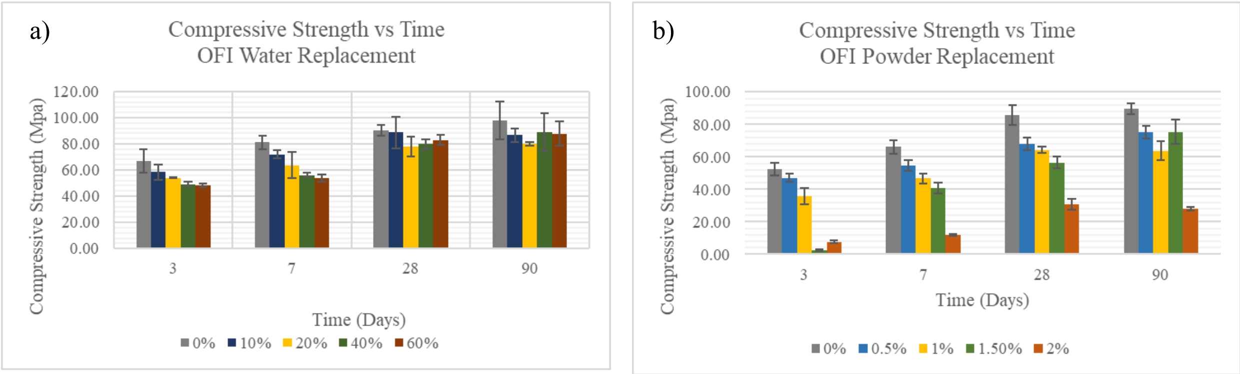

Figure 4. Graphs showing results for cement pastes compressive strength vs time for a) OFI water replacement and b) OFI Lyophilised Powder Replacement

3.2. *Mortar Hardened Properties*

3.2.1. *Ultrasonic Pulse Velocity*

Figure 5 presents the Ultrasonic Pulse Velocity(UPV) results obtained at 3,7,28 and 90 days for both types of replacement. Results obtained for the mortars containing OFI water replacement showed a percentage decrease of 7% in UPV in the early stage, with respect to the control samples at 3 days. This UPV decrease was reduced to 4% at 7 days, ultimately leading to an increase of 4.3% in UPV at 28 days. This increase was further maintained at 90 days. The highest increase in UPV was obtained for the highest OFI water replacement (60%).

Samples containing OFI powder replacements, showed similar trends to the OFI water replacements, with an initial decrease in UPV at 3days of testing and an increase in strength at 7 and 28 days of testing. In this case, the initial decrease in UPV was more evident and this is attributed to the higher OFI concentration.

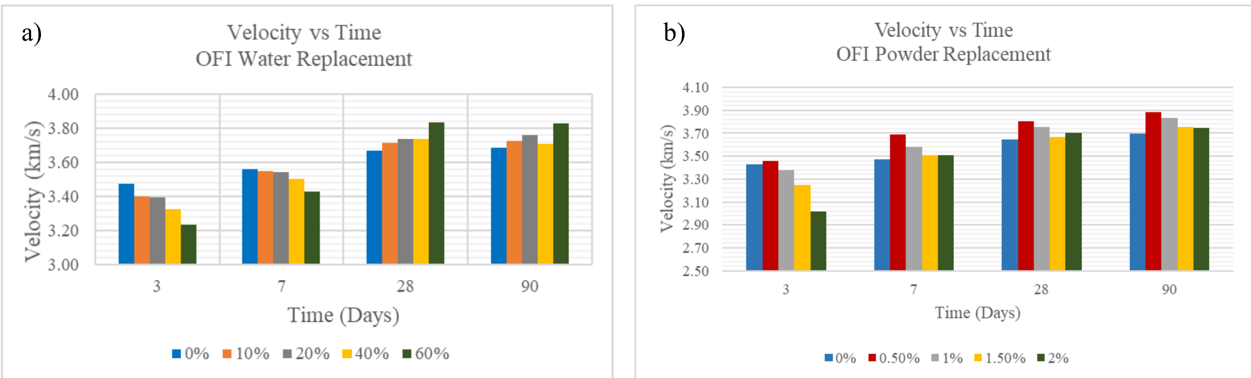

Figure 5. UPV vs time for a) OFI water replacement and b) OFI Lyophilised Powder Replacement

3.2.2. Compressive and Flexural Strength

Compressive strength results shown in Figures 6 obtained from the mortars containing OFI additions exhibited similar trends to the results obtained for Ultrasonic Pulse Velocity. The results obtained for the compression and flexural tests, show an initial reduction in strength for the mortar samples containing the higher percentage of OFI water replacement. The smaller percentage replacement showed equal or higher compressive and flexural strength results even at the early stages and this may be due to the fact that retardation was not much evident in these samples. However, at the 28 day of testing, the highest water percentage replacement (60%) showed the greatest increase in compressive and flexural strength from all the percentages with respect to the control samples.

In the case of the OFI powder replacements, similar results were obtained. In this case the initial decrease in strength was greatest for the highest percentage inclusion. The lowest inclusion of OFI powder (0.5-1%) registered the highest increase in compressive strength throughout the testing period.

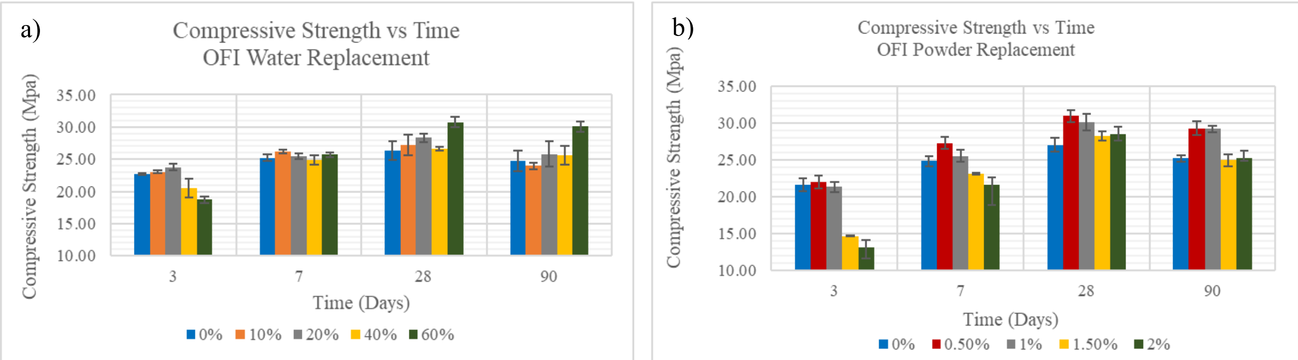

Figure 6. Graphs showing results for mortar samples, compressive strength vs time for a) OFI water replacement and b) OFI Lyophilised Powder Replacement

4. Conclusion

The use of *Opuntia ficus-indica* extracts showed promising results when used in cement pastes both as water and power replacements. Results for setting time indicate that *Opuntia ficus-indica extracts* can be used as retarding additive in both liquid and powder form, where an increase in both the initial and final setting times was recorded. Nevertheless, a relationship between adequate workability and retardation is crucial. Cement pastes with 0.5% and 1% OFI Powder replacements, exhibited prolonged setting time and therefore had a retardation effect, while still maintaining adequate workability. However, this increase in setting time negatively affected the compressive strength gain in cement pastes.

In the case of cement-based mortars, results indicated a general decrease in workability for increasing percentage replacement of both OFI powder and water replacements. Compressive strength results also showed an initial decrease in strength at 3 and 7 days due to the retardation. At 28 days increases in UPV, compressive and flexural strength were recorded for OFI replacements in both powder and liquid form. At certain percentages, this increase in strength surpassed that of the control mortar. This indicates that *opuntia ficus-indica* both in powder and liquid form is a potentially useful addition to enhance the hardened properties of cement-based mortars.

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