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Substitutive Effect of Cellulose Nanofibers for Cement in High Performance Concrete

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Abstract

Plain concrete has a very low tensile strength, a little amount of ductility, and little crack resistance. Internal microcracks are a natural component of concrete, primarily as a result of drying shrinkage, and when these microcracks spread, the concrete becomes brittle and has a weak tensile strength. Concrete's static and dynamic qualities will be enhanced by the addition of nanofibers, which will prevent the emergence of microcracks. In a study, nanocellulose fibers were used to strengthen High Performance Concrete (HPC) to improve durability. The benefits of alternative micro- and nano-fiber reinforcing methods could be achieved with fibers at a significantly lower cost. The impact of various reinforcing schemes on the processing variables and mechanical characteristics of a concrete mixture were empirically investigated. Notched-beam tests were conducted with crack-mouth opening displacement control in order to evaluate fracture energy under stable crack-growth conditions. According to preliminary data, adding 3% micro- and nano-fibers increased the fracture energy by more than 50% as compared to the unreinforced material with barely any changes to the processing method. Preliminary data show that, with hardly any modifications to the processing technique, the addition of 3% micro- and nano-fibers enhanced the fracture energy by more than 50% when compared to the unreinforced material.

Keywords

Nanocellulose fiber, Microcellulose fiber, High performance concrete, Notched beam test

Introduction

The distribution of cellulose fibers in concrete is three-dimensional, which effectively reduces the stress concentration at the tips of microcracks, weakens or eliminates the tensile stress brought on by dry shrinkage of concrete or mortar, and inhibits the emergence and growth of microcracks. The consistent distribution of cellulose fiber in concrete creates a support structure, prevents surface water precipitation and aggregate settlement, lessens bleeding and bleeding channels, and significantly reduces porosity. As a result, concrete now has much better impermeability. Cellulose fiber assists in absorbing the impact on concrete component function. Because fiber has a crack-resistance effect, it can effectively increase the impact resistance and toughness of concrete by preventing internal cracks from rapidly expanding when exposed to an impact load.

One kind of nanomaterial having high specific surface area, high strength, and high surface energy is nanocellulose. It can also be extracted from biomass resources and is nontoxic, innocuous, biocompatible, and environmentally beneficial. On the nanoscale, cellulose's surface groups exhibit high surface energy and binding activity, and they are modifiable through a variety of techniques. Nanocellulose is a great material for polymer reinforcement because of its high stiffness, low thermal expansion coefficient, and high elastic modulus. The idea of using cellulose crystals' high strength and stiffness in composite applications gave rise to the idea of using nanocellulose as reinforcement. Renewably sourced and reasonably priced, cellulose can regulate plastic shrinkage and boost concrete's strength. Because of its unique mechanical properties and high specific surface area, nanoscale cellulose has the potential to improve the strength of concrete.

Onuaguluchi et al. [1], the possibilities and restrictions of employing relatively small volume percentages of cellulose nanofibers (CNFs) utilized in cement pastes as reinforcing are examined in this research. Mejdoub et al. [2], the use of nano-fibrillated cellulose has increased the cement's hydration level. X-ray diffraction and Fourier Transform Infrared spectroscopy both supported this pattern. These studies demonstrate that the inclusion of nano-fibrillated cellulose increased cement hydration by enhancing the production of portlandite and calcium silicate gel, which is most likely the primary factor contributing to the noticeably increased compressive strength. Correia et al. [3], utilizing CNFs as reinforcement could improve particle packing and slow the rate at which cracks develop in nanoscale composites. Cement particles adhere to one another more tightly as a result of the increased specific surface area of CNFs. This study compared hybrid composites reinforced with 8% pulp and 1% nano-fibrillated cellulose to composites reinforced with only 9% pulp made by the extrusion technique.

Ultra-small structures with dimensions between micro- and nano-metres are employed in many different fields, such as sensor technology. To do this, lithographic techniques like photolithography are used to structure a resist. Materials systems with a size range of 1 to 100 nanometres are referred to as nanostructures. Electrons in a nanostructure are often allowed to travel in all directions in one dimension while being constrained in the other. Anything that falls between 1 nm and 999 nm in metric sizes is considered nanoscale. The formal limit of nanoscale is still 999 nm, although 100 nm is a widely accepted upper bound for nanomaterial size.

Jiao et al. [4], CNFs, which are renewable and naturally derived, are used to reinforce cement in this article. CNFs' impact on cement pastes' mechanical characteristics, degree of hydration, and microstructure has been researched. Akhlaghi et al. [5], in this study, the possible impacts of bacterial nanocellulose on the mechanical characteristics of cement mortar were investigated. To achieve this, bacterial nanocellulose (BNC) was used to make cement paste in two different ways: directly as powder and gel (0.1, 0.3, and 0.5 wt.%), and indirectly by coating polypropylene fibers with BNC. According to the findings, samples with BNC gel and powder had improved mechanical properties.

Using cellulose as a filler for a synthesis matrix or a natural starch matrix presents a great way to reduce the impact of non-biodegradable materials. It is the main component of plant cell walls, which aid in keeping plants rigid and erect. Plant sources, animal pellicle, and agricultural waste all contain it. In this research banana peel is used as a nanocellulose material. For the majority of applications, cellulose nanocrystals—which are mostly derived from naturally occurring cellulose fibers—serve as a sustainable and environmentally friendly material because they are renewable and biodegradable in nature. The contribution of the following research:

- To enhance the tensile strength nanocellulose and microcellulose fibers were used.
- Using a reactive powder concrete mixture, to determine how different reinforcing schemes affected processing variables and mechanical attributes.
- In order to evaluate fracture energy under stable crack-growth conditions, notched-beam tests with crack-mouth opening displacement (CMOD) control were conducted.
- Additionally, splitting tensile tests were carried out to contrast with beam-bending tests.
- Using high-resolution 3D imaging techniques to look at changes in damage paths and physical microstructures that result in increased toughness.

Nguyen et al. [6], this study's goal is to examine the mechanical and microstructure characteristics of hardened cement paste that contains Nylon 66 nanofibers. Xuan and Wang [7], in this article, they describe a multi-technique analysis of the macroscopic and microscopic features of materials with CNFs addition. Investigations were made into the samples' microstructure, mechanical characteristics, autogenous shrinkage, hydration kinetics, and durability. These are the outcomes: (1) CNFs promote later hydration while suppressing early hydration. Sun et al. [8], in order to optimize the distribution of steel fibers and the mechanical properties of UHPC, this study aims to provide a novel method by altering the matrix rheology with CNFs suspensions. It was discovered that the mechanical properties of UHPC and the dispersion of steel fibers were connected. The tests' findings showed that UHPC containing CNFs experienced multiple cracking as opposed to single cracking failure, along with an increase in tensile strength of 11 - 23%. Raso et al. [9], it has been done in this work to prepare and characterize novel cement mortars that have been reinforced with nano-fibrillated cellulose fibers. Brightson et al. [10], taking into account the final size of the reinforcement based on the strength of the mechanical treatment applied, the impact of the degree of fiber fibrillation on the mechanical performance of the resulting composites is described and analyzed. Du et al. [11], compared to normal concrete, UHPC has better workability, mechanical characteristics, and durability. However, some obstacles prevent UHPC from being used more widely. Brightson et al. [12], these challenges include inadequate flexural/tensile characteristics, significant autogenous shrinkage, limited workability for large-scale manufacture, and uncertain durability after concrete breaking. As a result, this research examines cutting-edge techniques for creating UHPC blends with enhanced characteristics.

Simões et al. [13], it was found that the tensile strength of the fiber, specifically, has a significant impact on the compressive strength growth, which generally increases with the reinforcing dosage. The type of reinforcement has an enormous impact on the load-displacement curves as well. Composite materials are better able to tolerate extreme deformations thanks to the addition of steel and polypropylene fibers. Because of their brittle nature, glass fibers have a diminished impact in this area. Akcay and Tasdemir [14], this study looked at the mechanical characteristics, fracture behavior, and mixture design of hybrid steel fiber reinforced self-compacting concretes. The combinations contained three distinct kinds of steel fibers, either with hooked ends or without (0.75 and 1.5 percent of the total volume of concrete). The workability of HSFRSCC was marginally decreased by adding additional fiber to the concretes, according to the findings of the slump flow, U-box, V-funnel, and J-ring tests. The shape of the fibers has the biggest impact on flowability. Although the final flowability was unaffected by the fibers, the flowability rate was reduced. Ramesh and Eswari [15], due to its remarkable qualities and low cost when compared to other natural fibers, basalt fiber has recently caught the attention of researchers in this respect. The focus of the current study is on the mechanical properties of concrete reinforced with basalt fibers and having a volume fraction of 0 - 2%. Rossi and Harrouche [16], the mechanical behavior of metal-fiber-reinforced concretes (MFRCs) is examined in this article in relation to the material's size and the size of the structure. It is demonstrated that the compressive and tensile strengths of MFRCs—used in reinforced concrete structures and having the workability of concrete—are consistently lower than those of conventional concrete, which is defined as the behavior seen before the stage of crack localization.

Albornoz-Palma et al. [17] have explained the qualities of the material requires an understanding of the morphology of CNFs, the rheological properties of their dispersions, and the corresponding relationships. While the intrinsic viscosity permits this relationship, the dynamic viscosity only partially reflects the behavior of the morphological qualities of the CNFs and does not offer trustworthy data that would allow these characteristics to be inferred with regard to the fibrillation process.

Materials and Method

HPC was strengthened with a mix of nanocellulose and microcellulose fibers to make a brittle material more resilient. Variable amounts of micro- and nano-cellulose fibers were added to the baseline HPC as reinforcement: 1%, 3%, and 5%, respectively, by weight of cement.

HPC

The preparation of the specimen is essential to the success of HPCs. Workability and processing constraints must be taken into consideration when creating mixtures because high-strength HPCs frequently contain only a small amount of water in the combination proportions. Reproducible processing techniques and mixing ratios are crucial and necessary. Varied mixture designs for different reinforcing levels of both micro- and nano-cellulose fibers were created in order to strike a balance between keeping a decent workability and achieving the lowest water-to-cement ratio possible. The effect of CNFs addition on early hydration of cement is examined by measuring water consistency and setting time using the Vicat test and it gives better form of concrete.

HPC mix proportion

Sand, Portland cement, silica fume, and silica flour were weighed out and layered in a mixing basin (Table 1). A simple stand mixer set to low speed was used to combine the dry ingredients for around 5 min. The superplasticizer and water were measured and combined while the dry ingredients were blending. Before adding them to the dry components, the superplasticizer and water were mixed for an unspecified amount of time. When cellulose reinforcement was used in the mixture design, it was added last.

The water, superplasticizer, and fiber combination then gradually added to the dry materials as the mixer continued to integrate them. When the wet portions were introduced, a few tiny balls immediately formed where the water touched the dry components. There were three main phases that the mixture went through. The first phase began with a moist appearance and continued for around two minutes. Three distinct dough-like clumps that formed during the second phase were its defining feature. Due to the material's stiffness, the second phase could be easily distinguished because it coincided with a rise in mixer torque. The mixture began to wipe the bowl on its own as this phase came to a conclusion. The 3rd phase's beginning was significant and provided an excellent idea of how the process would develop. Although the mixture's final stage had a consistency more similar to paste, it was still too thick to be poured directly from the bowl. The substance's rheology was slightly plastic and sticky. The mixture was agitated for a further 10 min to achieve a paste-like consistency, and it was then immediately cast into molds for test specimens.

Cylindrical and beam specimens were built for testing. For evaluating splitting tensile strength, 8 x 16 mm cylinders were created in compliance with ASTM specifications. The nominal dimensions of beam samples made for the flexural testing were 220 x 30 x 50 mm. The notch depth was determined as 13 mm, and the span is 180 mm.

Curing process of HPC

Three steps made up the HPC curing process. The samples were initially cured in a laboratory setting with moisture saturation for 7 days at room temperature. The samples were then maintained in a dry oven for 48 h at 90 °C. The specimens were completely cured and had attained their maximum power after this 13-day regimen.

Table 1: Mix proportion of HPC.

Material	Proportion
Water	0.20
Sand	0.97
Portland cement	1
Superplasticizer	0.017
Silica flour	0.27
Silica fume	0.40

Nanocellulose mix design

The Process Development Center at the University of Maine produced the nanocellulose fibers that were utilized to reinforce the baseline HPC. When the nanocellulose fibers were created, they were bonded in a water solution that had 3% fibers by weight. The amount of water utilized in each nano-reinforcement method was the same, but the amount required for the microcellulose fiber increased linearly. The 5% nanocellulose fiber mix design employed more superplasticizer than the 1% and 3% nanofiber mix designs. In contrast to 5% microfiber combination, a workable 5% mix was produced by increasing the superplasticizer. Table 2 displays the superplasticizer and water quantities for fiber-based nanocellulose reinforcement.

Microcellulose mix design

As anticipated, adding microcellulose fiber reinforcement altered the amount of water and superplasticizer needed to get the desired consistency. Microcellulose fibers were added to the base HPC at rates of 1%, 3%, and 5% of fiber reinforcement by the weight of cement, respectively. The modifications to base mixture needed to add microcellulose fibers are listed in table 3. A nearly linear relationship exists between the rise in fiber reinforcing and the rise in the water cement ratio. In a similar vein, the amount of superplasticizer required as fiber reinforcement increases is directly proportionate.

Laboratory testing of specimen

Testing three-point bending on notched beams and splitting tensile on 8 x 16 mm cylinders were the main objectives of this study. The fracture toughness of HPC at various levels of cellulose fiber reinforcement was assessed in order to compare the outcomes of each test to the performance of the control material.

Tensile tests for cylinder splitting

The specimen was crushed on its side during the indirect tension test at a rate of 0.08 mm/min till a crack began to spread throughout the specimen. The specimen was progressively released after the crack had formed. The unload sequence was taped in order to record the specimen's remaining energy.

Testing for three-point bending in notched beams

The three-point bending testing of the notched beams adhered to the test method for assessing fracture characteristics as described in the draft recommendations [7, 8] of the International Union of Testing and Research Laboratories for Materials and Structures. The test is run with the CMOD control and closed-loop control. The beam specimen may gradually degrade under this type of test setup. A linear variable displacement transducer (LVDT) was used in the studies to control the crack opening and keep the growth rate constant. Using aluminum mounting brackets that were firmly fastened to the beam specimen, the LVDT was installed across the notch. The specimen was loaded until a crack developed, then it was loaded again to allow the crack to widen out over time. According to the recommendations, the critical crack length (Ac), critical crack tip opening displacement (CTODc), critical stress intensity factor (Ks 1c), and fracture energy were calculated.

Results and Discussion

Tensile results in splitting

Calculating the fracture energy absorbed by the specimen was made possible by the splitting tensile tests carried out on cylinders 8 x 16 mm. Based on relative comparisons, several specimen types were compared, as shown in table 4 even though the calculation was size dependant and all the specimens evaluated were the same size.

Tensile splitting discussion

The average peak load of the 3% fiber cylinders was equal

Table 3: Quantities of superplasticizer and water for microcellulose fiber reinforcement.

Reinforcement	Water content	Change (%)	Superplasticizer (g)	Change (%)
None	0.2173	-	2.82	-
1% Microcellulose fiber	0.2416	11.4	3.32	18.1
3% Microcellulose fiber	0.3000	33.7	3.32	18.1
5% Microcellulose fiber	0.3600	61.3	3.92	40.2

Table 4: Fracture energy summary for tensile testing on splitting cylinders.

Reinforcement	Number of specimen	Peak load (N)	Fracture energy (mJ)
None	35	2052	92
1% Microcellulose fiber	37	1800	100
1 % Nanocellulose fiber	34	1800	100
3% Microcellulose fiber	42	2100	170
3% Nanocellulose fiber	32	1952	60
Hybrid	32	2400	84

Table 2: Quantities of superplasticizer and water for fiber reinforcement.

Reinforcement	Water content	Change (%)	Superplasticizer (g)	Change (%)
None	0.2173	-	2.82	-
1% Nanocellulose fiber	0.2416	11.4	3.32	18.1
3% Nanocellulose fiber	0.2416	11.4	3.32	18.1
5% Nanocellulose fiber	0.2416	11.4	3.92	40.2

to the peak load of the unreinforced specimens, showing that 3% cellulose fiber reinforcement was superior to alternative reinforcing approaches. Since the average peak load of the 3% microfiber cylinders was equivalent to the peak load of the unreinforced specimens, the findings of the cylinder tests showed that 3% nano- and micro-cellulose fiber reinforcement was superior to other reinforcing techniques. Due to a slight reduction in peak loads and only modest increases in fracture energy, 1% of reinforcement—micro and nano—did not seem to have any advantages.

Results of notched beam

Table 5 displays the outcomes of the notched-beam test. Critical effective crack length (Ac), one of the characteristics evaluated, was the most accurate predictor of increasing fracture toughness. Another crucial statistic was fracture energy (GF), which showed how much force was needed to cause the break to spread throughout the specimen. K1c and CTODc, the other two indices examined in this study, were also suggestive of fracture behavior but were deemed less significant.

The essential effective crack length differed very little amongst the various reinforcing procedures, according to the findings of the notched-beam testing. The reinforced specimens' average ac was 0.02 m, compared to 0.019 m for the notched beams that did not get any further reinforcement. The critical effective crack length served as a gauge for how challenging it was to initiate a crack in the specimen. It is possible that there were no fibers at the fracture tip because the crucial effective crack length did not alter when fiber reinforcements were added. They couldn't have been fully engaged before a fracture developed, so if one didn't already exist, they couldn't have prevented it from spreading. The crack's origin and growth through the specimen were measured by the fracture energy. As shown by GF, once the crack had formed, it was challenging for it to spread through the substance. More reinforcing led to an increase in GF as the fibers began to span the fracture and the specimen's ability to dissipate energy improved.

Figure 1 for notched three-point bend tests illustrates the load-deformation curves for unreinforced specimens and reinforced with 3% cellulose fiber. The region below the load-deformation diagram was used to compute the GF. There is additional room beneath the curve in the upgraded case. Greater surface area on a specimen with 3% cellulose fiber reinforcement may indicate that more energy was wasted during testing. In comparison to an unreinforced specimen, the GF for a 3% reinforced specimen was 50% higher. The hybrid somewhat increased the fracture energy (24% more than the unreinforced beams). The fracture performance of the HPC was not improved by the 1% micro- and nano-cellulose fiber specimens.

Beam deformation

According to studies and research, when concrete ages, its compressive strength will rise. The majority of studies were done to examine concrete's 28-day strength. In contrast to the long-term strength that it can develop with age, the strength in the 28th day is actually lower. There are numerous approaches that can be used to study how concrete strength changes with age. The total deformation of the concrete was analysed and briefly elaborated in table 6. Figure 2 explains the stress vs time of the analysed values. Since time and force are inversely correlated, force decreases with passing time which can be expressed in figure 3. Since time and momentum are directly inversely related, as time passes, momentum also increases. This value does not change when the acceleration is continuous.

The figure 4 shows load-deformation curves for unreinforced specimens and reinforced with cellulose fiber. It is parabolic in the compression zone, with zero at the top and its

Table 5: The reinforced notched-beam three-point bending tests fracture parameters in summary.

Reinforcement	AC (m)	K_{1c} (kPa-m$^{1/2}$)	$CTOD_C$ (m)	G_F (J/m^2)
Baseline	0.019	2400	0.01	60
1% Microcellulose fiber	0.022	2000	0.01	55
1 % Nanocellulose fiber	0.022	2400	0.01	58
3% Microcellulose fiber	0.022	1500	0.01	89
3% Nanocellulose fiber	0.019	1900	0.01	60
Hybrid	0.019	1700	0.01	74

Table 6: Total deformation of concrete.

Time (s)	Total deformation (mm)	Equivalent stress concrete (MPa)	Equivalent stress REBAR (MPa)	Equivalent elastic strain: CONCRETE (Max) (mm/mm)	Equivalent elastic strain: REBAR (Max) (mm/mm)	Force reaction (N)	[P] Force reaction (Total) (N)
10	5.175	85.735	224.72	3.08E-03	1.12E-03	-2.12E+05	2.12E+05
15	8.2802	137.31	359.9	4.93E-03	1.80E-03	3.39E+05	3.39E+05
20	10.35	171.5	449.52	6.16E-03	2.25E-03	-4.23E+05	4.23E+05
25	13.455	223.05	584.65	8.01E-03	2.92E-03	5.51E+05	5.51E+05
30	15.525	257.32	674.46	9.24E-03	3.37E-03	-6.35E+05	6.35E+05

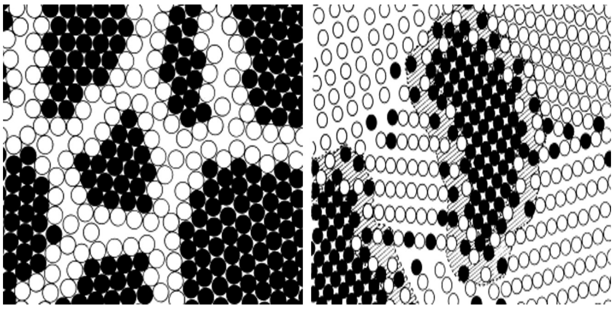

Figure 1: Nanostructure and microstructure of CNF.

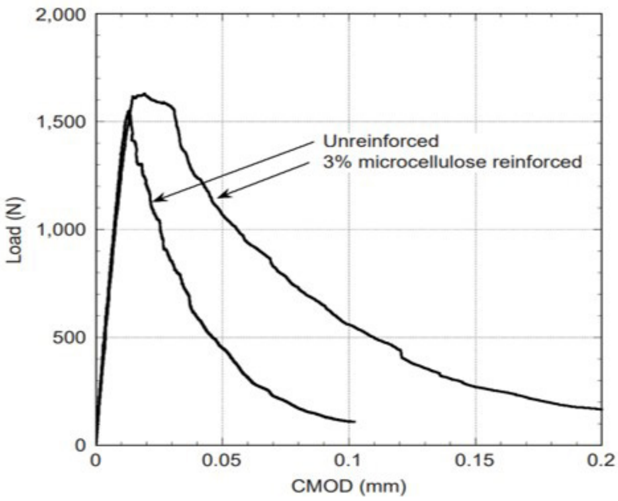

Figure 2: Typical load–deformation chart (CMOD - Crack Mouth Opening Displacement).

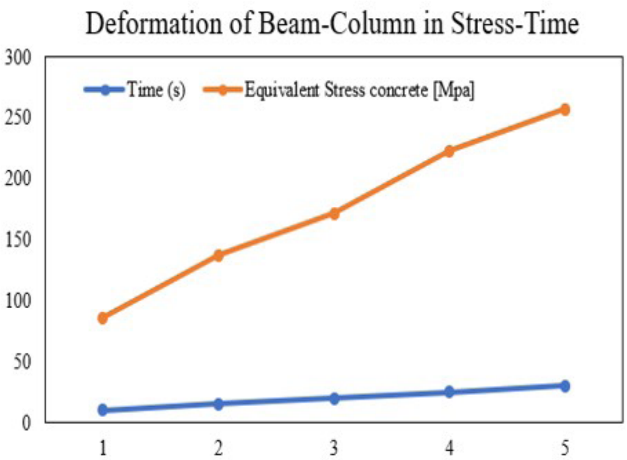

Figure 3: Deformation of beam-column in stress-time.

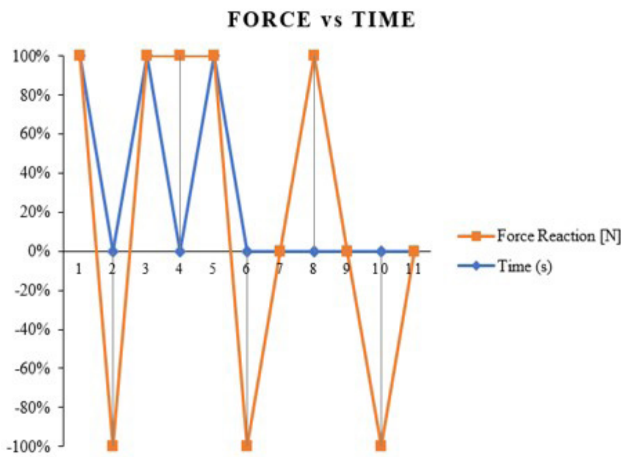

Figure 4: Force vs Time.

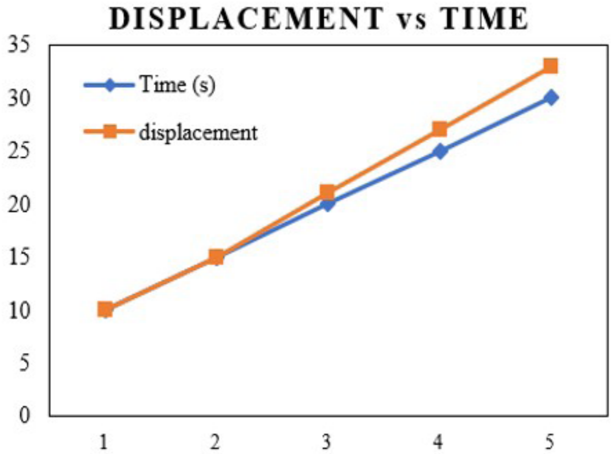

Figure 5: Displacement vs Time.

maximum along the neutral axis. Distance is the length of an object's path, whereas displacement is just the distance between an object's initial location and its ultimate location which can be explained in figure 4. Displacement is to describe a change in an object's position in relation to a reference frame. Figure 5 explains the displacement vs time of the analysed values.

Conclusion

The findings of this study show that adding cellulose fibers can enhance a HPC's fracture properties. The investigation's main goal was to ascertain how different cellulose-fiber reinforcing methods affected HPC's processability and fracture behavior. According to the investigation's findings, HPC can support up to 5% of cement weight in fiber reinforcement, 3% cellulose significantly increased fracture toughness, as demonstrated by the splitting tensile and notched-beam tests, which measure fracture energy. The small variation in crack length (Ac), revealed that the fibers may not have been positioned near the fracture tip, which would explain that they were ineffective at delaying crack initiation. The fibers, as can be seen in GF, were able to stop the cracks from propagating and turned active as they attempted to pierce the material. When 3% cellulose fiber reinforcements were added, the GF, of notched beams increased by 50% over the unreinforced specimens. The scope of the project is the creation of CNFs encourages the use of plant waste that is produced during food processing in agro industries.

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None.

Conflict of Interest

None.

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