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Comparison of the Effect of Nano Cellulosic Additives on the Rheological Parameters of Cement Paste

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ABSTRACT

In cementitious mixtures, parameters such as viscosity, yield stress and thixotropy must be controlled in order to design workability and flow properties. Especially in some special concrete applications, increasing viscosity and yield stress over time directly affect the quality of the hardened element. Here, cellulose nanocrystal and cellulose nanofiber were used in the cement paste in order to preserve the rheological properties initially designed and to keep the workability loss to a minimum. The rheological parameters of the mixtures containing cellulose nanocrystal and cellulose nanofiber additives in various proportions were determined by the Bingham model, and the thixotropy was determined by the area between the up and down curves of the shear rate-yield stress graphs. As a result of the study, the increase in yield stress was reduced by 79% and the increase in viscosity was reduced to 37% from the initial moment to 45 minutes. Moreover, the thixotropic property was improved with additives and at the end of 45 minutes, it was preserved at a maximum of 73%. It was determined that the cellulose nanofiber additive performed better than the nanocrystal additive.

Nano Selülozik Katkıların Çimento Hamurunun Reolojik Parametrelerine Etkisinin Karşılaştırılması

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ÖZ

Çimentolu karışımlarda, işlenebilirlik ve akış özelliklerinin tasarlanabilmesi için viskozite, akma gerilmesi ve tiksotropi gibi parametlerinin kontrol altına alınması gerekmektedir. Özellikle bazı özel beton uygulamalarında zamanla artan viskozite ve akma gerilmesi sertleşmiş elemanın kalitesini doğrudan etkilemektedir. Burada, başlangıçta tasarlanan reolojik özelliklerin zamanla korunabilmesi ve işlenebilirlik kaybının minimumda tutulabilmesi amacıyla, çimento pastasında selüloz nano kristal ve selüloz nanolif kullanılmıştır. Çeşitli oranlarda selüloz nano kristal ve selüloz nano lif katkısı içeren karışımların reolojik parametreleri Bingham modeli ile, tiksotropisi ise kesme hızı-akma gerilmesi grafiklerinin iniş ve çıkış eğrileri arasında kalan alan ile belirlenmiştir. Çalışma sonucunda, başlangıç anından 45 dakika sonrasına kadar akma gerilmesindeki artış %79, viskozitedeki artış ise %37'e kadar düşürülmüştür. Dahası, katkı ilaveleri ile tiksotropik özellik iyileştirilmiş ve 45 dakika sonunda en fazla %73 oranında korunabilmiştir. Selüloz nanolif katkısının, nano kristal katkısına göre daha iyi performans gösterdiği belirlenmiştir.

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1. Introduction

The rheological properties of fresh concrete/mortar have become more important today with the developing construction technologies such as self-compacting concrete (SCC), 3D printable concrete (3DCP), and underwater concrete (Petit et al., 2007; Tay et al., 2019). Fresh properties of concrete/mortar are critical to its strength, durability, and workability.

Yield stress and viscosity directly affect workability, so these are the two most important rheological parameters. Since the yield stress and viscosity of cement composites increases with time (Mostafa and Yahia, 2016; Kruger et al., 2019; Chen et al., 2020a; Liu et al., 2021, 2022), workability loss could be observed. The vibration applied during placement can greatly improve the workability of traditionally manufactured concrete (Felekoğlu and Sarikahya, 2008). However, vibration not be used in some specific concretes such as SCC and 3D concrete. Therefore for these specific applications have some particular rheological properties. For example, mixtures should preserve the initial slump level as much as possible in SCC and 3DCP and this can be achieved by controlling the yield stress and plastic viscosity increase (Felekoğlu and Sarikahya, 2008; Soltan and Li, 2018). In order for these special concretes to be applied the cement pastes in the mixtures also should be high thixotropic (Le et al., 2012; Paul et al., 2018; Liu et al., 2021).

Thixotropy is a system that is formed as a result of physical bonds when cement particles come into contact with each other for a certain period of time (Biricik and Mardani, 2022). In thixotropic systems, viscosity and yield stress decrease when deformation rate is applied, and when left to rest, viscosity and yield stress increase (Yuan et al., 2018; Navarrete et al., 2020). High thixotropy is demanded in SCC and 3DCP in order to exhibit the appropriate behavior in flow or resting time (Soltan and Li, 2018). The thixotropic area can be determined as the area between the up and down curves of the cement paste exposed to different shear rates (Quanji et al., 2014; Long et al., 2019a).

The following additives have been used in the literature and it has been observed that they increase thixotropy: silica fume, metakaolin, fly ash (Ahari et al., 2015; Yuan et al., 2018), attapulgite, nano calcium carbonate, nano silica (Yuan et al., 2018), limestone powder, re-dispersible polymer powder, and hydroxypropyl methylcellulose ether (Feng et al., 2022). Additives such as microcrystalline cellulose (Long et al., 2019b), hydroxypropyl methylcellulose, silica fume (Liu et al., 2021), metakaolin, and bentonite (Chen et al., 2020a; Liu et al., 2021), are used in the literature to adjust the viscosity and yield stress of the mixtures. In this study, cellulose nanofiber and cellulose nanocrystal additives obtained from cellulose, a renewable, environmentally friendly polymeric raw material, were used in order to increase thixotropy of mixtures and control the increment of viscosity and yield stress. In order to observe the changes in yield stresses and viscosities over time, the viscosity measurements of the mixtures with resting times of 0,15,30,45 minutes were made with a viscometer device, and the yield stresses of the cement pastes were determined in the Bingham model. The effects of the cellulose-based additives on yield stress, plastic viscosity and thixotropy were investigated.

1.1. Research Question

It is known that sustainable cellulose nanomaterials obtained from different sources are effective in providing the desired rheological properties for concrete.

In this study, answers were sought to the following questions:

Could the increase in viscosity and yield stress of the cement paste with time be controlled by using cellulose nanocrystals and cellulose nanofibers?

How these additives effect thixotropic behavior?

Which additive performs better in which situations?

2. Material and Method

2.1. Preparation of mixtures

The additives mainly affect the flow behavior of the cement paste without changing the composition or behavior of the aggregates (Ferraris et al., 2001), so in this study the tests were performed on the cement paste. This study aimed to control the rise in viscosity and yield stress of cement paste over time. CEM I 42.5 N Ordinary Portland Cement (OPC) was used in the preparation ofpastes. A new generation polycarboxylate-based superplasticizer with a high water reduction capacity and modified phosphate based setting retarder additive were used.

Commercially provided cellulose nanocrystal (CNC) and laboratory-produced cellulose nanofiber (CNF) were used for viscosity regulation in the mixtures. The plant-derived CNC used in the study hada white color, a width of 10-20 nm, a length of 300-900 nm, and a density of 1,49 g/cm^3. CNCs, which were homogenized by mixing with an ultrasonic homogenizer for 1 hour in mixing water, were then included in the mixture. Another viscosity-regulating additive used in this study was CNF obtained from a green alga, Cladophora sp. The hydrochloric acid hydrolysis method used in Türk et al. were used to obtain CNFs from Cladophora sp. Accordingly, algae were first purified from soil, minerals, proteins, pigments, and unwanted materials. Then, the purified samples were exposed to sound waves with a sonicator for 1 hour to ensure the separation of the fibers and the formation of nanofibers. As a result of the aforementioned process, cellulose nanofibers with a diameter of 15-20 nm and a length of 1500-2500 nm were obtained (Türk et al., 2022). Before the cellulose nanofibers were included in the mixtures, they were mixed and homogenized with the mixing water for 1 hour with an ultrasonic homogenizer.

The mixture without any viscosity modifying additives was named as a reference. Between 0,025-%0,075, CNF and CNC were included in the reference mixture. The mixtures produced within the scope of the study are presented in Table 1.

Table 1. Mix proportions

Sample Name	OPC (kg/m^3)	Water (kg/m^3)	Superplasticizer (%)*	Retarder (%)*	CNC/CNF (%)*
Reference	800	280	1	0,5	0
CNC 1	800	280	1	0,5	0,025 CNC
CNC 2	800	280	1	0,5	0,05 CNC
CNC 3	800	280	1	0,5	0,075 CNC
CNF 1	800	280	1	0,5	0,025 CNF
CNF 2	800	280	1	0,5	0,05 CNF
CNF 3	800	280	1	0,5	0,075 CNF

*by weight of binder

2.2. Method

The mixtures given in Table 1 were mixed for one minute. Viscosity measurements of cement pastes with resting periods of 0,15,30 and 45 minutes from the preparation of the mixture were made. Brookfield DV2T model viscometer was used for viscosity measurements. A rotating apparatus with a diameter of 11,76 mm and a length of 33,02 mm and a sample chamber with a sample volume of 10,4 ml were used. (Figure 2.1).

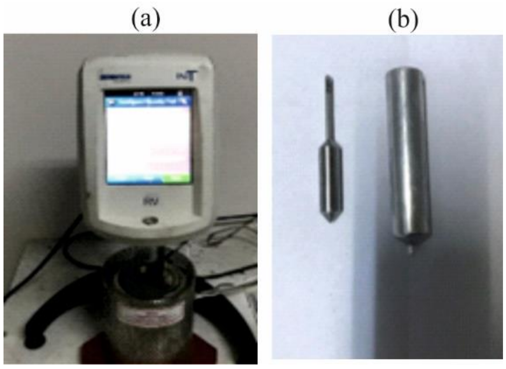

Figure 2.1. Viscometer device (a), spindle and sample chamber (b)

The prepared cement pastes were pre-sheared at 30 s-1 for 30 seconds. This pre-shear aimed to ensure that each batch of mortar has the same initial shear state during the rheological analysis. After this pre-shear, it was waited for 30 seconds for the mixture to stabilize. Then, within 90 seconds, the shear rate increased in the range of 5 s^{-1} to 65 s^{-1} (5 s^{-1} intervals) and decreased. The shear stress corresponding to each shear rate was recorded during the measurement. The Bingham model is widely used when examining the rheologic parameters of fresh concrete (Yuan et al., 2017; Chen et al., 2020a; Jiao et al., 2021; Liu et al., 2021). For the determination of yield stress and plastic viscosity, the values of the down curve and the Bingham model given in Equation 2.1 were used.

$$\tau = \tau_0 + \mu p \text{ x } \gamma \quad (2.1)$$

Here, τ (Pa) is the shear stress, τ_0 (Pa) is the yield stress, γ (s-1) is the shear rate, and μp (Pa.s) is the plastic viscosity. The yield stress of the cement paste was determined by making linear regression with the data obtained from the measurement at various shear rates. As a result of the measurements, a comparison was made between the yield stress and plastic viscosities.

Thixotropy was measured as the area between the up and down curves of the shear rate shear stress graph. The results that changed with the effect of the additives were compared.

3. Result and Discussion

3.1. Bingham models

Bingham models prepared depending on the resting time of mixtures containing CNF are presented in Figure 3.1, and mixtures containing CNC are presented in Figure 3.2. In general, yield stresses and viscosities of the samples containing additives remained above the reference sample at all resting times. In addition, the yield stress and viscosity values increased with the increase in the amount of additive at all resting times. This is likely due to the agglomeration of CNF and CNC. Cellulosic nanofibers form a network structure. As larger forces are required to break or align these structures, yield stress increases (Cao et al., 2015). In addition, the increase in the yield stress of cement pastes due to the increase in the rate of additives may also be due to their hydrophilic nature and their excessive holding capacity and holding the mixing water (Muhammad Salman et al., 2021; Liang et al., 2022).

It is clear from the measurement results taken immediately after the preparation of the mixture (Figure 3.1a), that the yield stress and viscosity of the sample with the highest CNF increased by %240 and %175, respectively, compared to the reference sample. However, after 45 minutes (Figure 3.1d), these rates of increase decreased to %65 and %23, respectively. A similar situation existedfor mixtures containing CNC. In the measurement taken immediately after the preparation of the mixture (Figure 3.2a), the yield stress and viscosity of the sample with the highest CNC increased by %176 and %135, respectively, compared to the reference sample. However, at 45 minutes (Figure 3.2d), these rates of increase decreased to %69 and %32, respectively. The effect of additives on hydration could explain this situation due to the adsorption of CNF and CNC on the cement particles, a delay in hydration happens as a result of the diminish within the accessible surface region of the cement particles to take part within the hydration response (Cao et al., 2016; Flores et al., 2017; Liang et al., 2022).The reference sample continued to hydrate at its usual rate from the moment of mixing until the 45th minute, so the yield stress and viscosity of the mixture increased continuously. However, the increase in yield stress of samples containing additives over time could be associated with flocculation and cement hydration (Reiter et al., 2018; Chen et al., 2020b).Therefore, the rate of increase in viscosity and yield stress slowed down due to the hydration delay in samples containing additives. In this way, the increase in viscosity and yield stress was controlled, and the open-time of the mixture was extended.

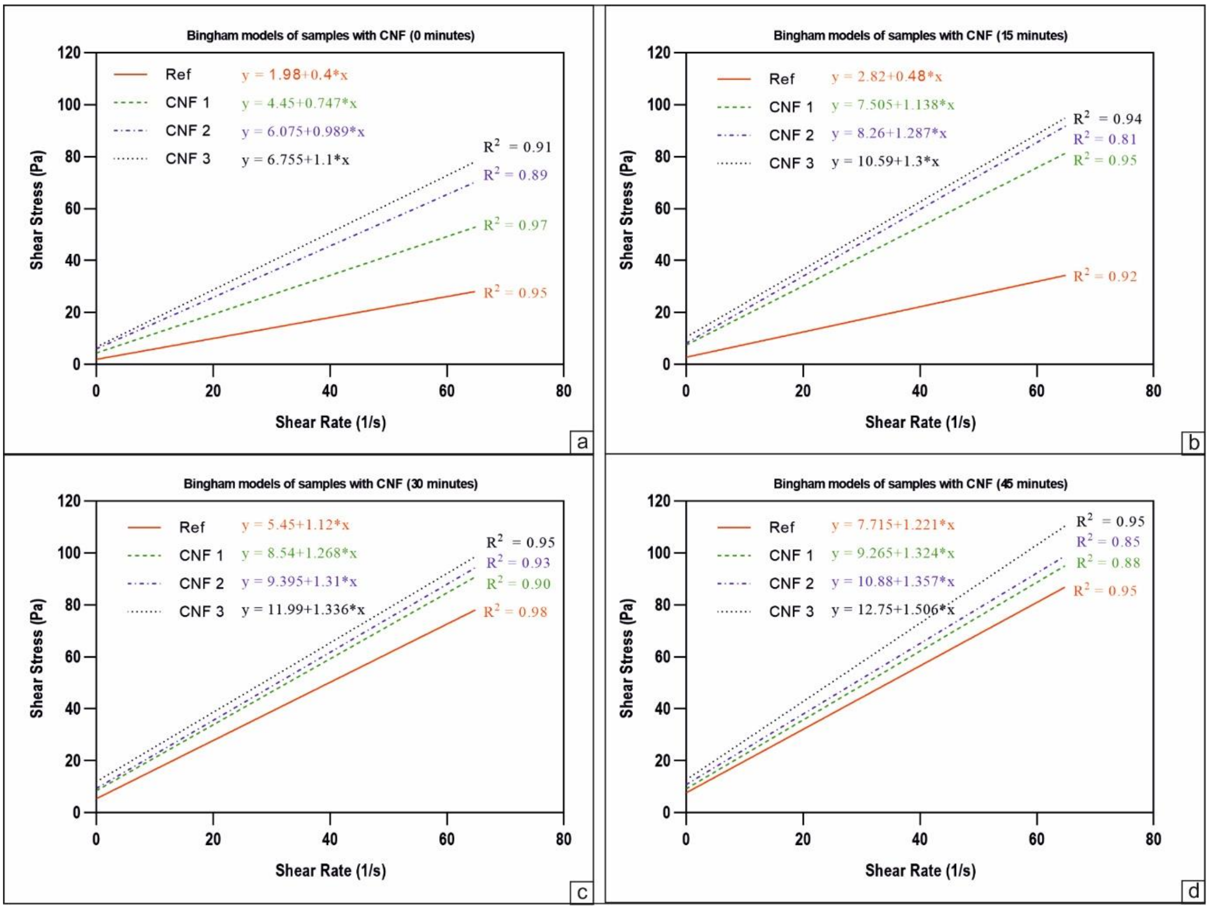

Figure 3.1. The Bingham model fitting results of the declining section of the shear rate for CNF, resting time (a) 0 minutes, (b) 15 minutes, (c) 30 minutes, (d) 45 minutes

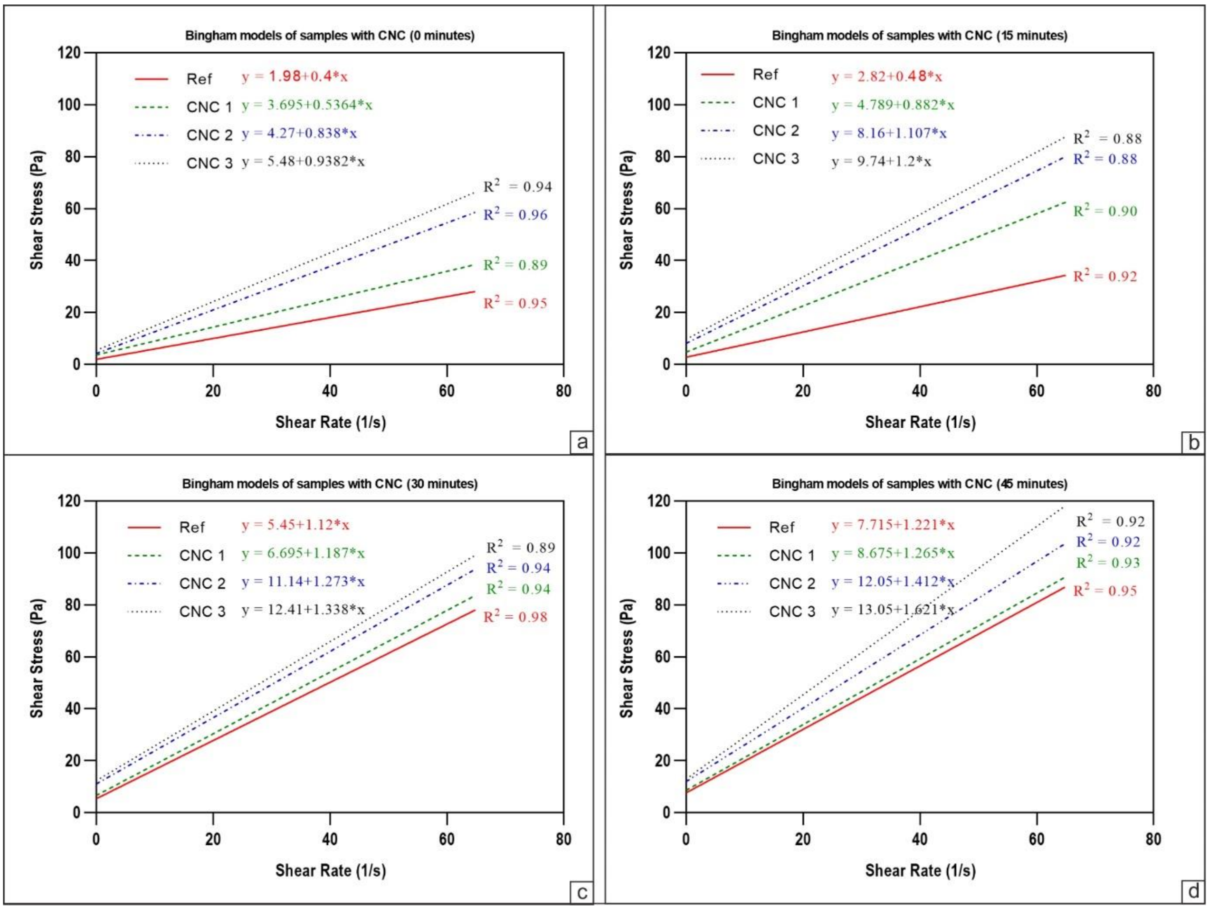

Figure 3.2. The Bingham model fitting results of the declining section of the shear rate for CNC, resting time (a) 0 minutes, (b) 15 minutes, (c) 30 minutes, (d) 45 minutes

3.2. Thixotropy

For the thixotropy evaluations, the mixture containing the median value of 0,05 was selected. In Figure 3.3, the shear rate-shear stress graph and thixotropic areas of reference, CNF 2 and CNC 2 mixtures that have 0,15,30,45 minute resting time are presented. As seen in the figure 3.3 the thixotropy value for the cement paste increases appreciably with the addition of CNF and CNC, this indicating an improvement in the thixotropy. It is noteworthy that pastes with CNF exhibited more improvement in thixotropy than pastes with CNC.

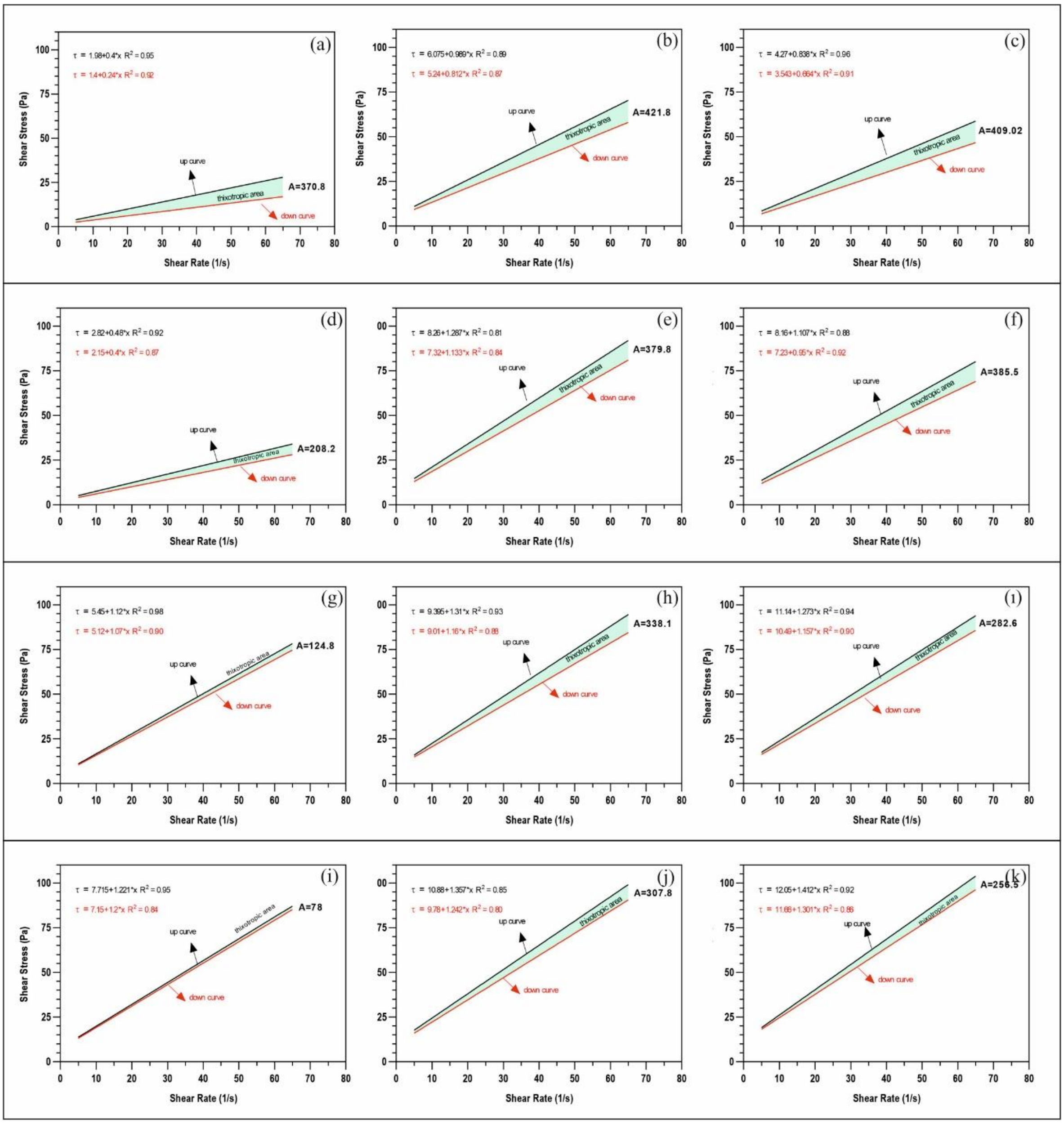

Figure 3.3. The thixotropic area of cement pastes, 0 minute (a) ref, (b) CNF2, (c) CNC2, 15 minutes (d) ref, (e) CNF2, (f)CNC2, 30 minutes (g) ref, (h)CNF2, (ı) CNC2, 45 minutes (i) ref, (j) CNF2, (k) CNC2.

The thixotropic area percentages preserved over time from the first mixing moment are given in Figure 3.4. The reference sample retained only %21 of its initial thixotropy 45 minutes after the initial mixing, while the sample containing nanofibers retained %73 and the sample containing nanocrystals %62. It is understood that the nanofiber additive is more effective in the preservation of thixotropy over time. These results confirm that the nanofibers align in the flow direction, facilitating the flow since shear stress is applied, and entanglement with each other during the resting period, forming a complex structure (Nassiri et al., 2021).

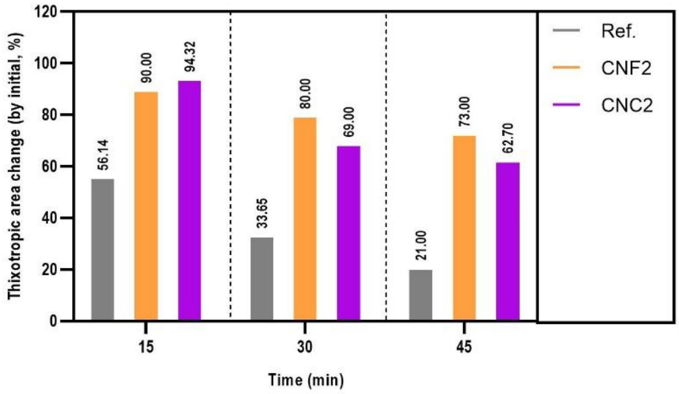

Figure 3.4. The thixotropic area preserved over time

3.3. Comparison of additives

The yield stress and viscosity increases of the mixtures from the first mixing moment are presented systematically in Figures 3.5 and 3.6 It was understood from this that the yield stress of the reference sample increased by %42 in 15 minutes, %175 in 30 minutes, and %290 in 45 minutes. However, the increase rates in samples containing additives were not that high. For example, the sample containing %0,05 CNF increased by %36 in 15 minutes, %55 in 30 minutes, and %79 in 45 minutes. When CNC and CNF additives are compared, CNF generally increased yield stress and plastic viscosity more than CNC. CNFs are physically much longer than CNCs and are often entangled. Due to this shape, they create a restrictive effect on the dispersion of cement particles. Since the fibers form a network, it becomes difficult to break or align them, thus increasing the yield stress (Gwon and Shin, 2021). CNCs, on the other hand, are smaller and mobile particles, so they do not restrict the movement of cement particles as much as CNFs (Nassiri et al., 2021). This explains why the yield stresses of mixtures containing CNC are lower than the yield stresses of mixtures containing CNF.

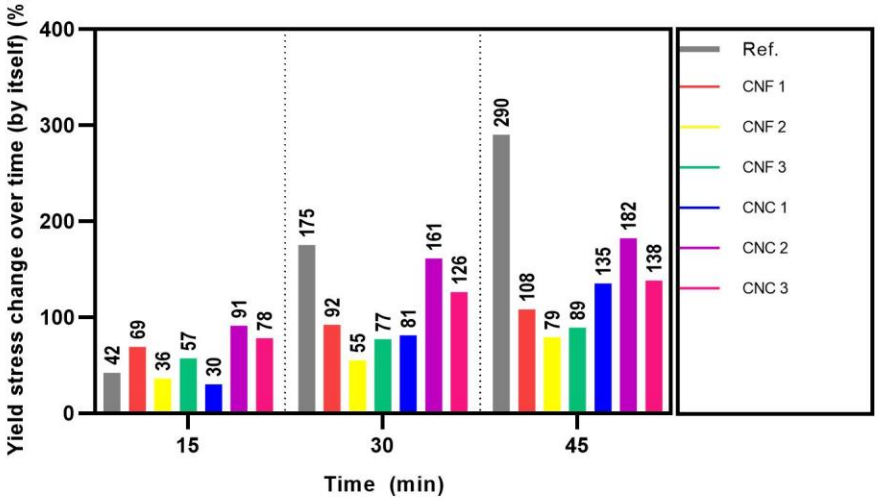

Figure 3.5. Percentage increase in yield stress of mixtures over time

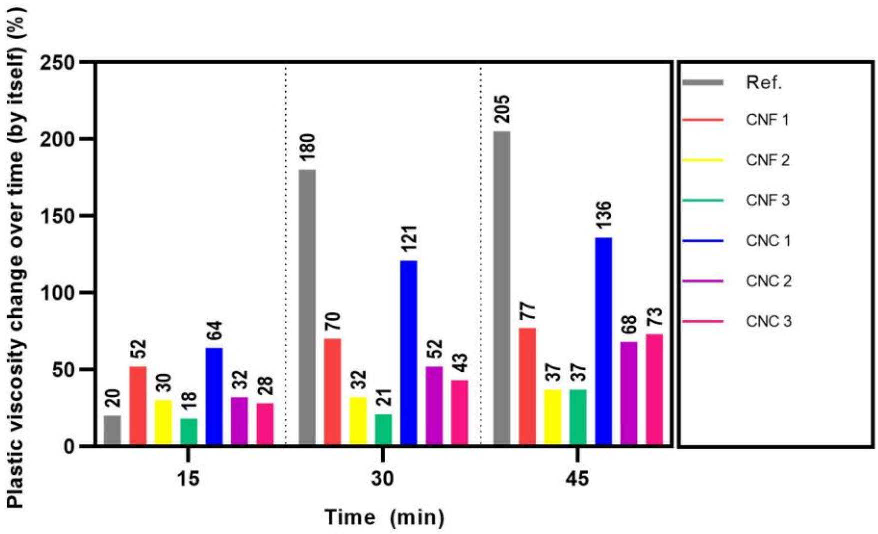

Figure 3.6. Percentage increase in viscosity of mixtures over time

4. Conclusions

In this study, which investigated the effects of two different cellulosic additives in nano size on the viscosity and yield stress of cement paste, the following conclusions were reached.

- CNF and CNC additives increased the viscosity yield stress of the mixtures depending on the amount of use.
- The use of CNF and CNC in concrete and mortar applications, where rheological parameters such as yield stress and viscosity must be controlled, improves performance. Considering the desired rheological parameters in 3DCP, it was thought that the use of CNF and CNC in the mixtures can be increased open-time by controlling the increase in viscosity and yield stress.
- When comparing CNF and CNC, using the same amount of additives, CNF increased yield stress and viscosity more than CNC. However, considering the variation of rheological parameters with time, the yield stress and viscosity increase rate of the mixture were lower when CNF was used. Therefore, using CNF in mixtures is more advantageous than CNC.
- Both types of additives are highly effective in preserving thixotropy. However, CCF additive is more effective than CNC.

Statement of Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author's Contributions

The authors contributed equally to this work.

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