SYNTHESIS OF NANO FIBRILLATED CELLULOSE BLENDS AND THEIR REHOLOGICAL BEHAVIOUR USING DIFFERENT MIXING TECHNIQUES

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Abstract: The purpose of this study was to reduce the viscosity of Nano fibrillated cellulose by mixing it with a non-ionic polymer such as poly vinyl alcohol and maintaining the dry solid content at the same level. An aqueous solution of poly vinyl alcohol was prepared and it was mixed with Nano fibrillated cellulose at three different concentrations. These were 0.08%, 0.1% and 0.12 %. The effect of different polymer concentrations on the rheology of Nano fibrillated cellulose was investigated. Modular compact rheometer 300 was used in order to obtain rheological data. Two different results were generated. The first one was the shear viscosity versus shear rate and the second one was the complex viscosity versus mixing time. Dispermat VL was used for the mixing of Nano fibrillated cellulose and polyvinyl alcohol. Two impellers with different geometry were used for mixing purpose. Double milling impeller was used with beads. 48 hours of mixing was carried out with dispersion impeller whereas for double milling impeller 5 hours mixing was used. An optical microscope was used in order to study the morphology of mixed samples. Obtained results showed that the viscosity of Nano fibrillated cellulose was reduced at all concentrations of poly vinyl alcohol where as an intermediate concentration of polyvinyl alcohol i.e., 0.1% was most effective compared to the other two concentrations. Moreover, mixing with dispersion impeller showed better results in terms of viscosity reduction as compared to double milling impeller.

Keywords: Nano fibrillated cellulose; Poly vinyl alcohol; Modular compact rheometer; Mixing impeller; Rheology

1. Introduction

Nano fibrillated Cellulose (NFC) [1] has been a great deal of attention from many years owing to its abundance [2], strength [3], flexibility [4], their renewable [5] and environmentally feasible nature [6], outstanding mechanical properties [7] and therefore, applied in various industries such as foods [8], cosmetics [9], paints [10], paper [11] and nonwoven textiles [12]. Cellulose can be extracted from the natural fibers. There are two main families of nano sized particles. The first one is composed of nano crystals and the other one is nanofibrilled cellulose [13]. The term nano is used to designate nano meter scale items (10-9 m) [14]. Since the introduction and the concept of nanotechnology given by Richard Feynman in 1959 [15] there has been an increasing interest from the scientific community to work with such kind of materials [16]. Due to its barrier properties as well as oxygen permeability, NFC films were studied that was made from the bleached spruce sulfite pulp [17]. The use of cellulose nano particles as reinforcements in nanocomposites is a relatively new area of interest [18].

As far as the production of nano scale fibers is concerned by means of mechanical disintegration process cellulose micro fibrils were extracted from wood cell by Herrick and Tubark back in 1983 [19]. It can be considered as a cellulosic material, which is primarily composed of high-volume cellulose, degraded and expanded in surface area [20] that can be obtained by a process known as to be homogenization [20].

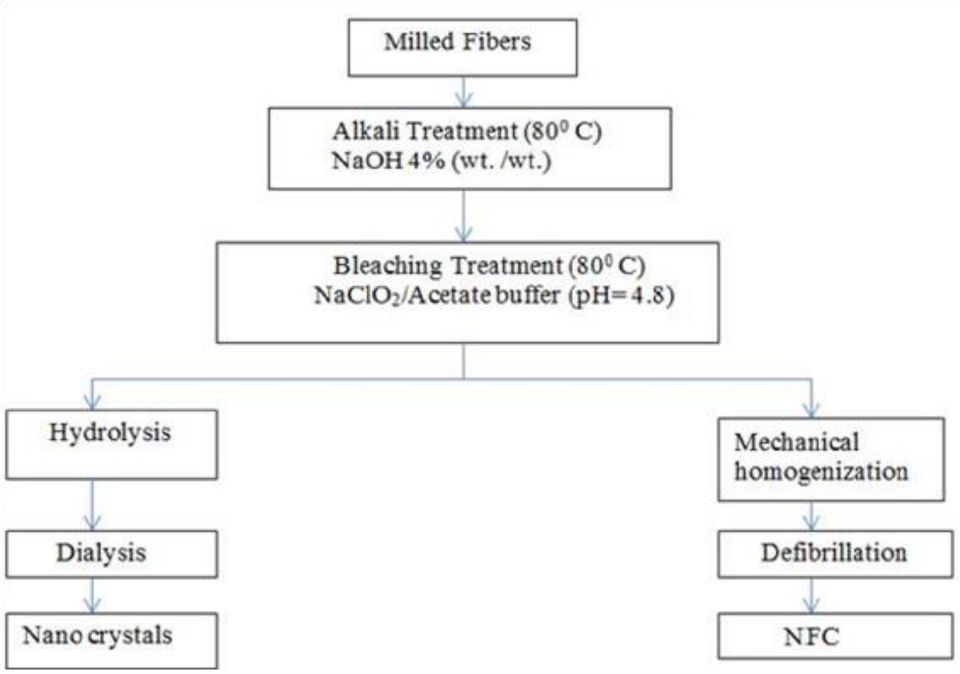

Figure 1. shows various steps involve in the production of both Nano crystals and NFC

The properties of both the cellulose whiskers and nano fibrillated cellulose are different from that of conventional cellulose which is straight chain [21]. The dimensions of cellulose nano fibrils are in the range of 10 to 100 nm [22]. The characterization by SEM of softwood pulp after 10 passes through a homogenizer revealed that NFC consists of both crystalline and amorphous domains [23] having an alternative structure in relation to one and other. While compared with cellulose whiskers one more prominent difference amongst their structure is that the NFC presents a web like structure [24].

To describe the rheology of polymers [25] the entanglement model is used [26]. In this model it is presumed that the polymer forms a shape of spaghetti [27]. In a polymer melt or in a solution long molecule entangle themselves loosely with others several times. Without any external load the polymer shows the shape of a three-dimensional coil [28]. when shear process [29] occurs i.e., shear rate is applied the molecules present in the polymer are more or less oriented in a shear direction. As result of disentanglement of molecules, the flow resistance decreases [30]. When oriented to high degree the chains may even disentangle themselves completely. As a result, the single molecules are no longer in touch of each other [31]. This concept can be used to describe the flow of shear thinning fluid and an ideal viscous fluid. Nano fibrillated cellulose shows a shear thinning behavior i.e., its viscosity decreases with increased shear rate [32]. The shear thinning curve of NFC [33] can be distinctively divided into three zones which are a no shear structure known as to be yield stress, a high shear structure called as high viscosity and a low shear structure i.e., low viscosity [34]. Shear thinning curve of NFC starts with a yield stress and no shear structure is present [35]. Higher concentrations of NFC show higher yield stress and higher viscosities [36]. At higher concentrations of NFC, the creation of network among the fibrils is stronger resulting in a higher viscosity of NFC. Apart from the well-known fact that the viscosity decreases with increased temperature the de swelling of fibers which increase the fibers and fibrils mobility also decreased the viscosity of NFC [37].

In order to reduce the degree of non-uniformity or gradient of a property in a system such as concentration, viscosity and temperature the process of mixing is used. The phenomenon of mixing is carried out by moving material from one region to another [38]. To obtain uniform mixture it is customary to understand how liquids move in the vessel. Two important requirements must be fulfilled during the mixing process such as the presence of bulk or convective flow [39] so that no dead zones should exist and a zone of intensive or high shear mixing in which the inhomogeneity are broken down [40].

Flow patterns are very much dependent upon the type of impeller being used in the vessel [41]. Efficiency of the mixing equipment and product quality are also influential in determining the flow patterns existing in the vessel. Flow pattern for the single phase both for the Newtonian and non-Newtonian fluids [42] have been extensively studied using turbine impellers and propellers [43]. Close clearance impellers are able to reach the far corners rather than relying on the turbulent momentum transport in highly viscous systems. There are three types of mixing flow patterns that are axial, radial and high efficiency impellers [44].

Mill base mixing using the dispersion process [45] itself can be divided into three steps. These are wetting of the surface of the solid particles by the fluid components of the mill base, mechanical breakdown of the associated particles and generation of smaller particles during dispersion. Areas of high and low pressure are formed when the vanes of the disc are moved through the mill base at a high velocity. For large particles a smashing impact must be considered that are being hit by the edges and the surfaces of the vanes [46]. Fast movement of the blade facilitates the shear that builds up on the surfaces of the disc in which the dispersion takes place [47]. The distance between the lower surface of the disc and the bottom of the container in which dispersion is being executed determines the amount of shear stress. At high rotational speed of impeller are used more mechanical power is being introduced into the mill base [48]. Maximum dispersion could be achieved with the highest possible mechanical power input but with only laminar flow. In order to determine the mechanical power input following formula is used $P = 2\ \pi n M$, where P = mechanical power input, n = rotational speed of the impeller and M = momentum (Torque) [49]. In mill base operations when the solid matter is difficult to disperse dispersion with the dissolver is insufficient. In this regard high speed bead mills have acquired particular acceptance due to their ability to process a wide variety of solid materials [50].

2. Materials and Methods

NFC STP with initial concentration of 2%

2.1 Alcotex 72.5 polyvinyl alcohol

Alcotex 72.5 is a medium molecular weight, partially hydrolyzed polyvinyl alcohol with a medium hydrolysis level of 72%. It was provided by synthomer international technology individual service. It is soluble in both hot and cold water. Polyvinyl alcohol is a non-ionic copolymer. Total dry solid content of PVA is above 95%. On industrial scale it has numerous applications such as formulating the reprographic coatings as well as specialized adhesives.

2.1 Preparation of PVA solution

Buch and Holm’s HB4D digital water bath was used to prepare the solution of alcotex 72.5 in hot water at a temperature of above 800 C. The dry solid content of alcotex were first determined (see appendix I). The solution concentration was 10 %. This was done in order to obtain the homogenized solution of Alcotex in water. The temperature was set to 850 C and the agitation was carried out at 600 rpm. The solution was mixed for 1 hour and then allowed to cool down at room temperature. Three different quantities of 10% solution of alcotex were used in the solution (see appendix I).

2.2 Preparation of NFC blends

Blends of Nano fibrillated cellulose and nonionic polymer Alcotex 72.5 were used for mixing purpose. Dry solid content of NFC STP was determined. Depending on the capacity of the mixer total weight of the NFC suspension mixed with PVA was 700 grams (see appendix 1 for the composition of the solution).

2.3 Mixing of the solution

VMA Getzmann’s dispermat VL shown in figure 17 was used for the mixing of NFC suspension and PVA. The mixing was carried out using two impellers having different geometry.

Figure 2. vacuum dissolver dispermat VL

2.4 Mixing with a dispersion impeller

Different mixing conditions were employed for these two impellers. The mixing time was 48 hours and the revolution speed was1000 rpm. Samples were taken out after every 10 second and then at 4, 8, 24, 28 and 48 hours of mixing. It is important to note that the samples were taken out from the same batch. Initial weight of the sample was 700 grams. The sample weight was 20 grams. If large amount of sample is withdrawn every time, then the weight of the sample in the mixer would be very low, so for this reason only 20 grams of sample was taken out every time.

Figure 3. Dispersion impeller with a varying diameter ranging from 30-60 mm

2.5 Mixing with a double milling impeller

Double milling impeller shown in figure 18a was used with beads. The solution to bead ratio was 1:5. Weight of one bead was approximately 0.08 grams. For bead mill the total weight of the solution was 600 grams and the weight of the beads was 100 grams. Mixing for bead mill was carried out for 5 hours. The samples were drawn at every 10 second and then at 1,2,3,4 and 5 hours of complete mixing. The sample weight was again 20 grams. Beads were isolated from the sample every time it was subject for rheological measurement. Every time sample was taken out beads were also withdrawn from the mixer in order to maintain the solution to beads ratio i.e. 1:5.

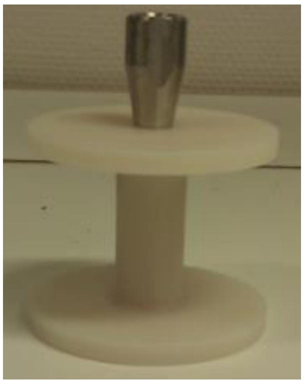

Figure 4. Double milling impeller with a varying diameter 70 mm

2.7 Dry solid content

Dry solid content was determined for every sample (see appendix I for the calculations). The samples were allowed to be dried in the oven at 105 ^{0}C for maximum one hour. Afterwards they were placed in a desiccator for 45 minutes in order to maintain room temperature.

2.8 Rheological Study

Paar Physica MCR 300 SN 477918; FW2.23 rheometer with universal software US 200 as shown in figure 20 was used in order to obtain rheological data. Parallel plate measuring system was first setup. After that the initialization of the machine was carried out. Zero gap was done. PP 50 measuring system was used and TEK 150P-C measuring cell was used. The measuring gap was set at 0.1mm. For viscosity profile measurements the maximum shear rate was 615.01 1/s and maximum shear stress was 4584 Pa. for complex viscosity the maximum shear rate was 2618 1/s and maximum shear stress was 6111,55 Pa. Temperature of the plate for the samples was set to 20º C. The number of data points that generated was 50 for each reading. Each sample was subjected twice for the measurement. In order to determine the complex viscosity oscillatory test was performed that examines all kinds of viscoelastic behaviors. These tests are known as to be dynamic mechanical analysis. In order to determine the complex viscosity amplitude, sweep test was performed with a constant frequency of 1 Hz and varying amplitude γ 0.1%.

Figure 5. Paar Physica Modular Compact Rheometer 300 with universal software US 200

2.9 Morphology

Olympus BX51 soft imaging system was used to obtain microscopic view of the samples in order to understand the morphology of the solutions with a magnification of 20X. All samples were 10% diluted.

2.10 Centrifugation

Hettich Universal 320 R centrifugation equipment was used in order to centrifuge all samples. After mixing NFC with PVA the samples were collected in centrifugation tubes and then were placed in the centrifugation equipment. 6 samples were placed at one time for 5 minutes. After that the pure water was decanted from the tubes and solid contents as well as rheological data were measured from the decanted samples. Centrifugation was carried out at room temperature and the number of revolutions was set at 3000 rev/min.

3. Results

3.1 Viscosity Profile

Figure 6 shows the viscosity profile of NFC as a function of shear rate. The concentration of NFC in the solution was 1.5 %. Mixing was conducted for 48 hours and the rheological data was obtained at every 10 seconds and then at 4, 8, 24, 28 and 48 hours of mixing. Results showed that with increased shear rate the viscosity of NFC decreased. Fluids exhibiting such kind of trend are known as to be shearing thinning fluids [28]. Figure 20 shows that the viscosity is highest at 10 seconds of mixing. As and when the time for mixing was increased the viscosity gradually decreased. The viscosity profile at 4 hour of mixing was lower as compared to 10 seconds of mixing. After 48 hours of complete mixing the viscosity was at its lowest value as compared to 10 seconds and 4,8,24 and 28 hours of mixing. Here it is important to note that the reduction in viscosity was not due to the presence of polymer it's with the time hence more mechanical power was induced that produced more shear stress on the fluid breaking its long chain and hence reducing the viscosity.

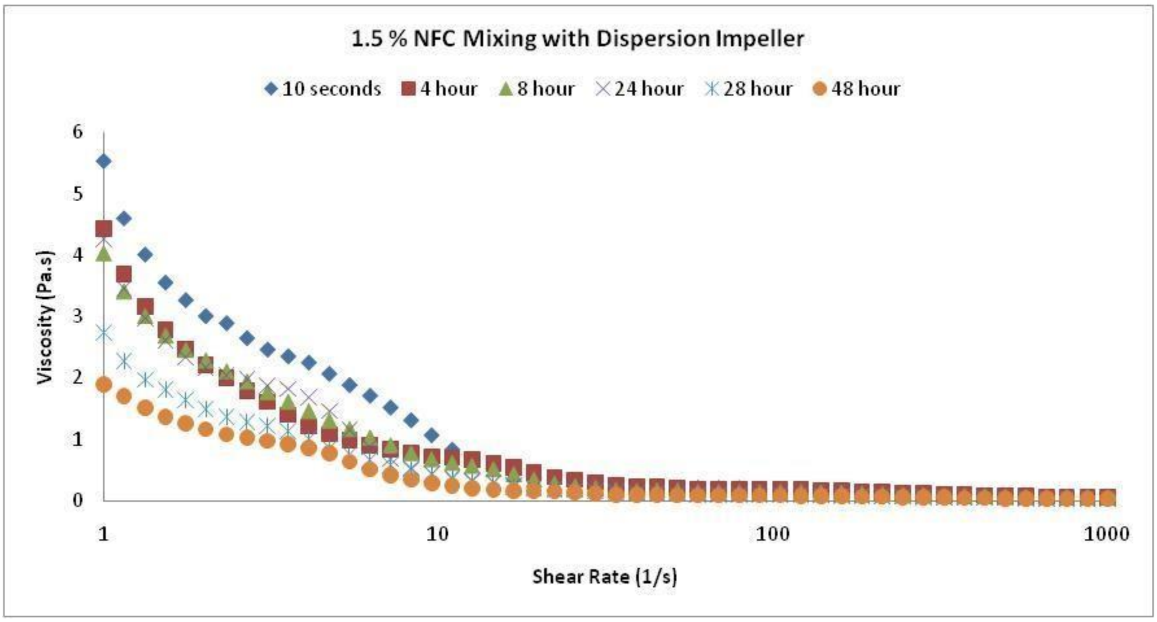

Figure 6. Viscosity as a function of shear rate for 1.5% NFC solution after mixing with a dispersion impeller for 10 seconds, 4-hour, 8-hour, 24-hour, 28 hour and 48 hours.

Figure 7 shows the viscosity as function of shear rate mixed with a double milling impeller. Beads were used in order to grind NFC.1:5 beads to solution ratio were maintained. The rheological data obtained showed that there has not been any significant change in the viscosity of NFC i.e. the viscosity remained same from 10 seconds to 5 hours to complete mixing. This trend was quite different from the one in which mixing was conducted for 48 hours with dispersion impeller. The reason for no change in viscosity could be due to impeller type as double milling impeller was used for mixing generating lower shear rate as compared to the other impeller.

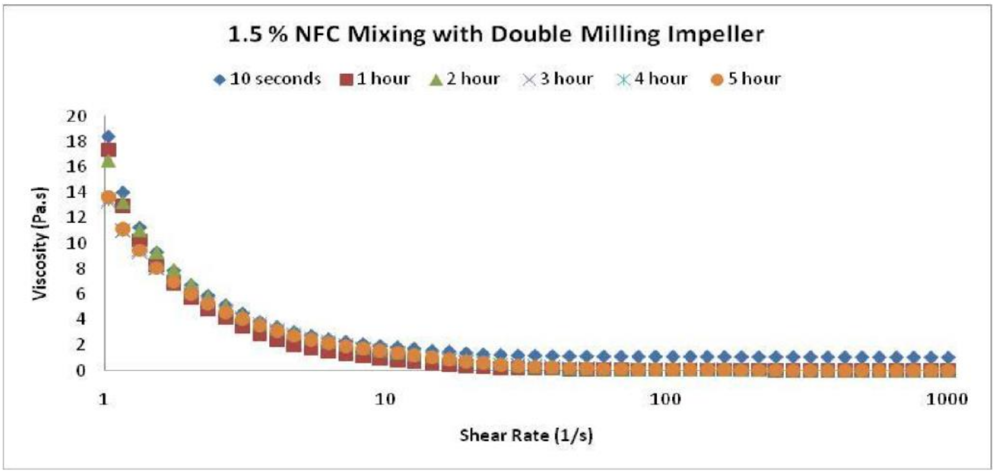

Figure 7. Viscosity as a function of shear rate for 1.5% NFC solution after mixing with a double milling impeller for 10 seconds, 1-hour, 2-hour, 3-hour, 4-hour and 5-hours.

Figure 8 shows the viscosity as a function of shear rate for 1.5% NFC with 0.1% PVA solution after mixing with a dispersion impeller. The behavior of NFC mixed with PVA was the same as that of pure NFC i.e. the viscosity decreased with the increased shear stress. Hence the NFC blend also showed the shear thinning behavior. Results showed that the viscosity was being decreased with increased mixing time with one exception that the viscosity at 28 hours was higher as compared to all other mixing times. In order to decrease the viscosity of NFC the adsorption of PVA on the surface of NFC has to be of irreversible type that is it should not detach itself from the surface of NFC. At 28 hours of mixing the adsorption would have been of reversible type as a result PVA remained in the solution that increased the viscosity of NFC. From 28 hour to 48 hour of mixing the viscosity reduced appreciably and remained at its lowest value as compared to 0 seconds as well as 4,8,24 and 28 hours of mixing

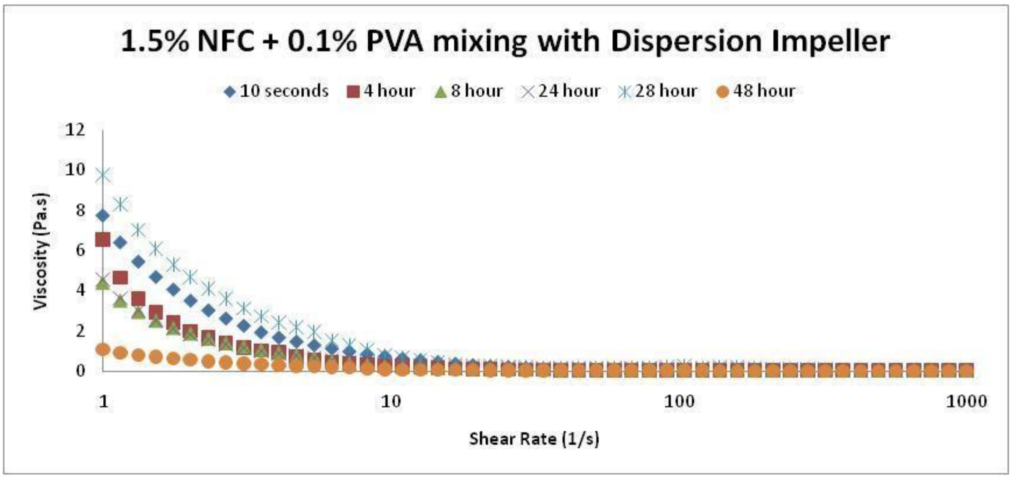

Figure 8. Viscosity as a function of shear rate for 1.5% NFC with 0.1% PVA solution after mixing with a dispersion impeller for 10 seconds, 4 hour, 8 hour, 24 hour, 28 hour and 48 hour

Figure 9 shows the viscosity as a function of shear rate for 1.5% NFC with 0.1% PVA solution after mixing with a double milling impeller. Samples were drawn at 10 seconds and then at 1,2,3,4 and 5 hours of mixing and were subjected for rheological measurements. At the start of the mixing the viscosity i.e., at 10 seconds viscosity of NFC blend as compared to 1, 2, and 3 hour of mixing was lower. After 4 hours of mixing the viscosity of NFC blend decreased as compared to 10 second of mixing. The difference in viscosity between 2 hour and 3 hour of mixing is lower. After complete mixing of 5 hours the viscosity was lowest as compared to all other mixing times.

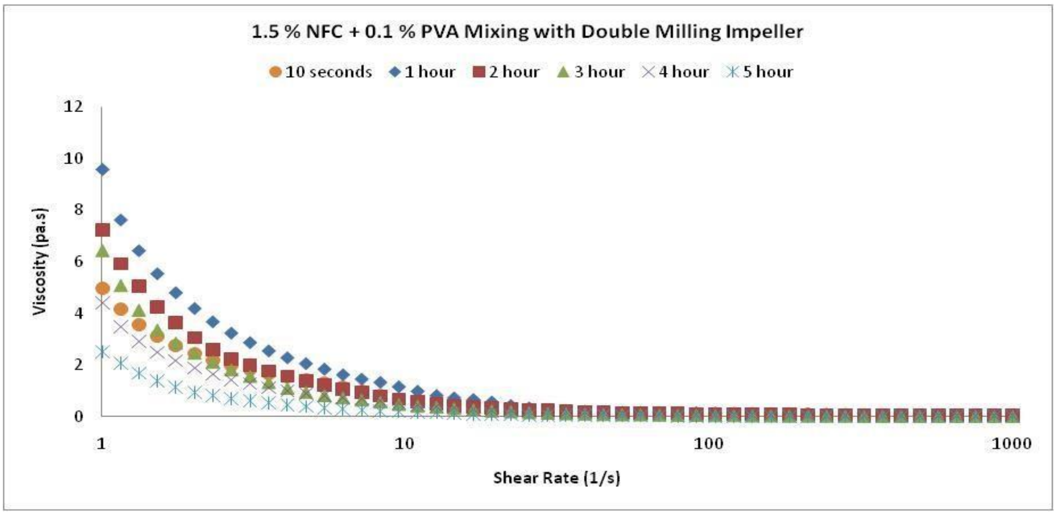

Figure 9. the viscosity as a function of shear rate for 1.5% NFC with 0.1% PVA solution after mixing with a double milling impeller for 10 seconds, 1 hour, 2-hour, 3-hour, 4 hour and 5 hours

Figure 10 shows the viscosity as a function of shear rate for all compositions used in the experimental work. Mixing was done with a dispersion impeller. Viscosity profile of pure NFC as received with 2% dry slid content, 1.5 % NFC and NFC mixed with PVA at all compositions is shown in this graph. The purpose of this thesis was to reduce the viscosity of NFC by mixing it with PVA at three different concentrations. Results showed that the viscosity of NFC cellulose was reduced either by processing the NFC alone or by mixing it with PVA. It also showed that the difference in the viscosity profile of 1.5 % and NFC mixed with PVA was not that much whereas the viscosity of 1.5% mixed with 0.1% PVA lowest as compared to the other compositions. On the other hand, at 0.12% concentration of PVA in the solution the viscosity was slightly higher as compared to the other ones indicating us the limitations. This could be due to the fact that at higher concentration of PVA the mobility of the molecules was reduced as a result the fluid becomes more viscous which increased the viscosity of NFC.

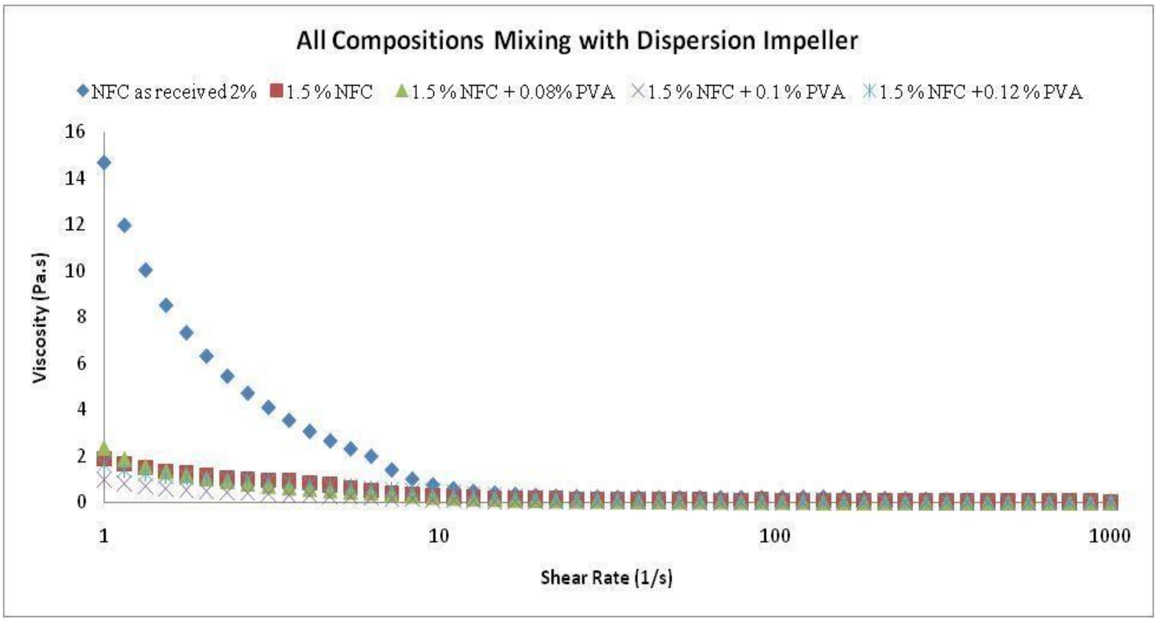

Figure 10. the viscosity as a function of shear rate for all compositions of PVA after mixing with a dispersion impeller for complete mixing of 48 hours

Figure 11 shows the viscosity as a function of shear rate for all compositions after mixing with a double milling impeller. The viscosity of pure NFC reduced when it is mixed with PVA at all concentrations. The viscosity of 1.5 % NFC that was processed alone in dispermat showed same values of viscosity as compared to the pure NFC. This indicated that the use of beads was not effective in reducing the viscosity of NFC alone whereas the viscosity was reduced significantly when it was mixed with PVA. Comparing all three concentrations of PVA again showed the same trend as in the dispersion impeller i.e., the viscosity of 0.12% PVA was higher than the other two concentrations and the viscosity of 0.08% PVA and 0.1% PVA was almost at same value

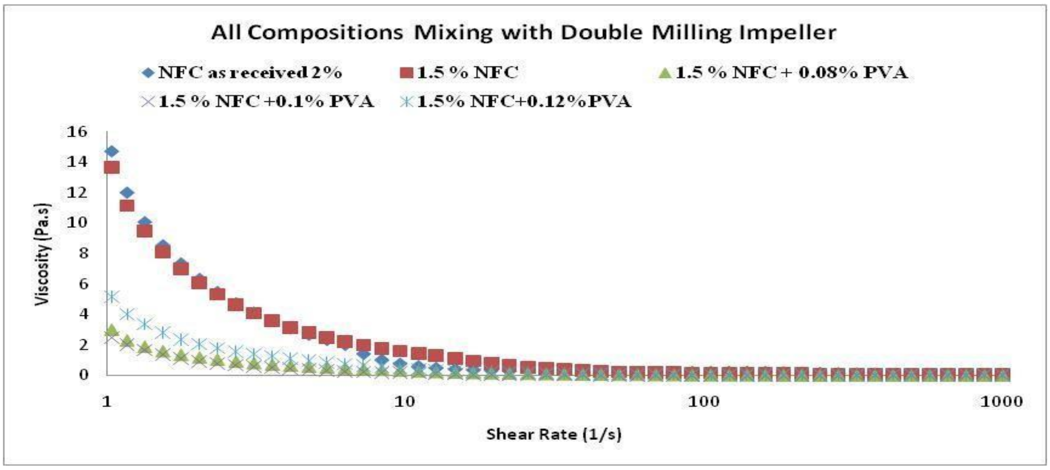

Figure 11. the viscosity as a function of shear rate for all compositions of PVA after mixing with a double milling impeller for complete mixing of 5 hours

Figure 12 shows the viscosity as a function of shear rate for all centrifuged samples after complete mixing with both dispersion and double milling impeller. Centrifugation of the samples was done in order to observe whether we could be able to achieve the same or lower viscosity as compared to the pure NFC with higher dry solid content. Before centrifugation the solid contents of the samples after complete mixing were around 1.5%. After centrifugation dry solid content rose to 4% (see figure 42).The results showed that the NFC mixed with 0.12% PVA in a double milling impeller has the highest viscosity as compared to all other compositions. The viscosity of 0.08 % PVA when mixed with both dispersion and double milling impeller was almost equal to the viscosity of Pure NFC as received. The dry solid content of these two compositions were higher than the pure NFC.

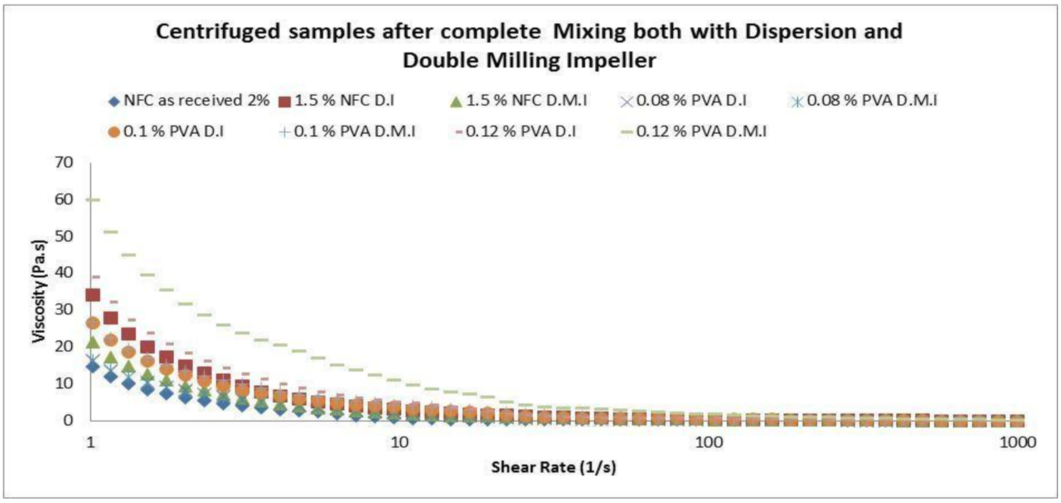

Figure 12 the viscosity as a function of shear rate for all centrifuged samples composition after mixing with both dispersion and double milling impeller

3.2 Complex viscosity

Figure 13 shows the complex viscosity as a function of mixing time for 1.5% NFC solution after mixing with a dispersion impeller. Rheological data for complex viscosity was obtained at the same hours for which shear viscosity data was attained. Results showed that the complex viscosity after 48 hours was lowest as compared to the other ones. The only peculiar behavior observed was that the viscosity after 28 hours was higher as compared to the 24 hours of mixing. Error bars shown in all the graphs represents the 95% confidence interval.

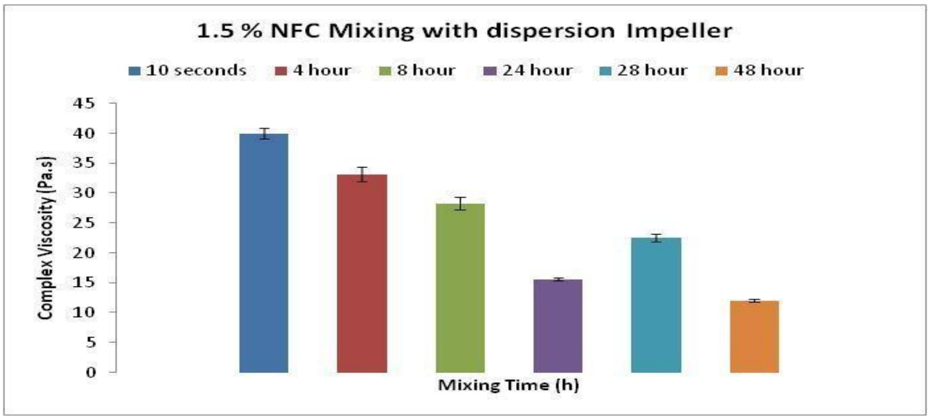

Figure 13. The complex viscosity as a function of mixing time for 1.5% NFC solution after mixing with a dispersion impeller for 10 seconds, 4 hour, 8 hour, 24 hour, 28 hour and 48 hour

Figure 14 shows the complex viscosity as a function of mixing time for 1.5% NFC mixed with a double milling impeller. The conditions for oscillatory tests were same with a constant frequency of 1 Hz. The mixing time was 5 hours. The results showed that with increased mixing time the viscosity of NFC has reduced. From 10 seconds to 1 hour of mixing the reduction of viscosity is not so much whereas after 2 two hours of mixing the reduction in viscosity is more appreciable. After complete mixing of 5 hours the complex viscosity was at its lowest value as compared to the other ones. Figure showed that the viscosity was higher at 3 hours of mixing at then it gradually decreased. The mechanical power being inducted into the impeller depends upon the number of revolutions and the torque produced [19]. The number of revolutions was kept constant whereas the torque changed with mixing time. Hence the increase in viscosity could be related to the fact that with changing torque the mechanical power of the impeller also changed hence the complex viscosity was higher at 3 hours as compared to 2 hours.

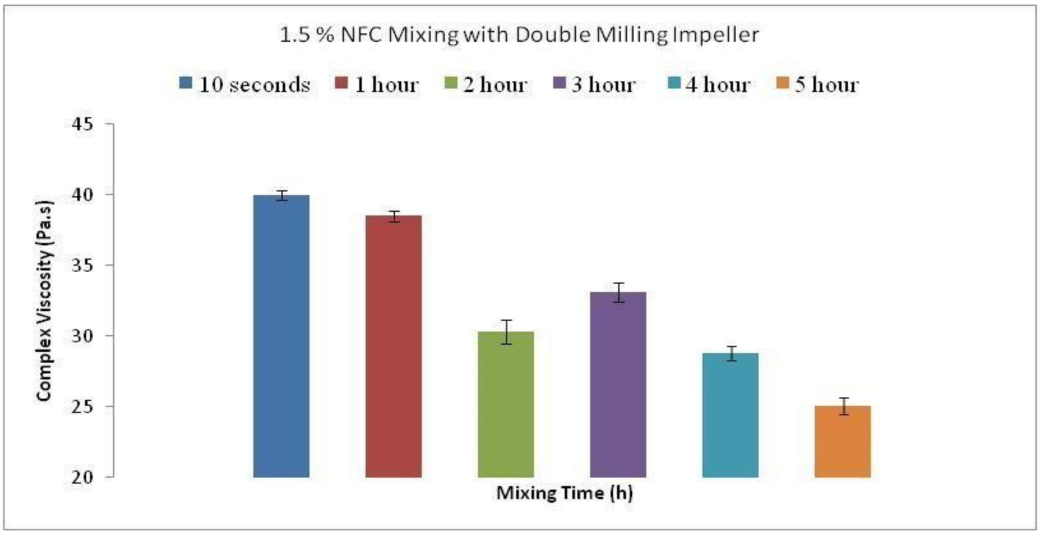

Figure 14. the complex viscosity as a function of mixing time for 1.5% NFC solution after mixing with a double milling impeller for 10 seconds, 1 hour, 2 hour, 3 hour, 4 hour and 5 hour

Figure 15 shows the complex viscosity of NFC/PVA blends a function of mixing time. The composition of PVA in the solution was 0.1%. The complex viscosity decreased with increasing mixing time. At 10 seconds of mixing the value of complex viscosity it highest as compared to 4, 8, 24, 28 and 48hours of mixing. After complete mixing the viscosity was lowest and the difference between the complex viscosity at 0 and 48 hour was appreciable. Figure also showed that the complex viscosity was more or less same for 4 hour to 28 hour of mixing. Reduction in viscosity was due to the reason that with increased mixing time more and PVA adsorbed onto the surface of cellulose hence reducing its viscosity.

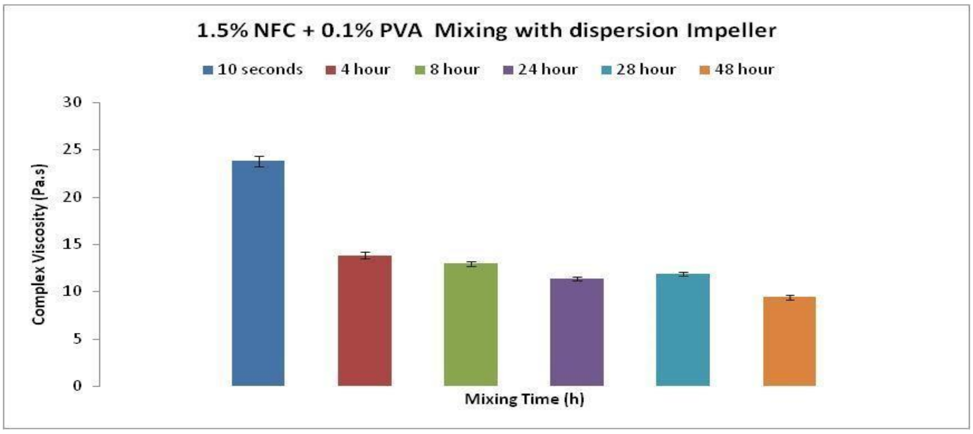

Figure 15. The complex viscosity as a function of mixing time for 1.5% NFC with 0.1% PVA solution after mixing with a dispersion impeller for 10 seconds, 4 hour, 8 hour, 24 hour, 28 hour and 48 hour

Figure 16 shows the complex viscosity of NFC/PVA blend as a function of mixing time. Complex viscosity while mixing with a dispersion impeller decreased with increased mixing time. The difference in complex viscosity between 0 and 1 hour of mixing was not significant whereas after complete mixing of 5 hours there was a clear difference in viscosity.

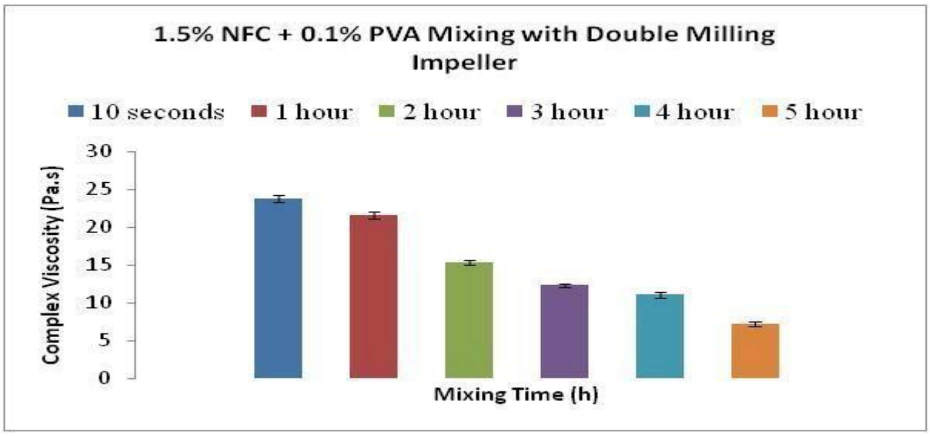

Figure 16. The complex viscosity as a function of mixing time for 1.5% NFC with 0.1% PVA solution after mixing with a double milling impeller for 10 seconds, 1 hour, 2 hour, 3 hour, 4 hour and 5 hour

Figure 17 shows the complex viscosity of all compositions used in the experimental work mixed with a dispersion impeller. It showed an extensive comparison amongst all the compositions of NFC/PVA. The concentration of NFC in the solution was constant i.e. 1.5% whereas PVA was used in three different concentrations i.e. 0.08%, 0.1% and 0.12%. Results showed that complex viscosity of pure NFC as received with 2% dry solid content decreased when it was mixed at three different concentrations of PVA. The complex viscosity of 1.5% NFC without mixing with PVA was lower than the complex viscosity of pure NFC as received

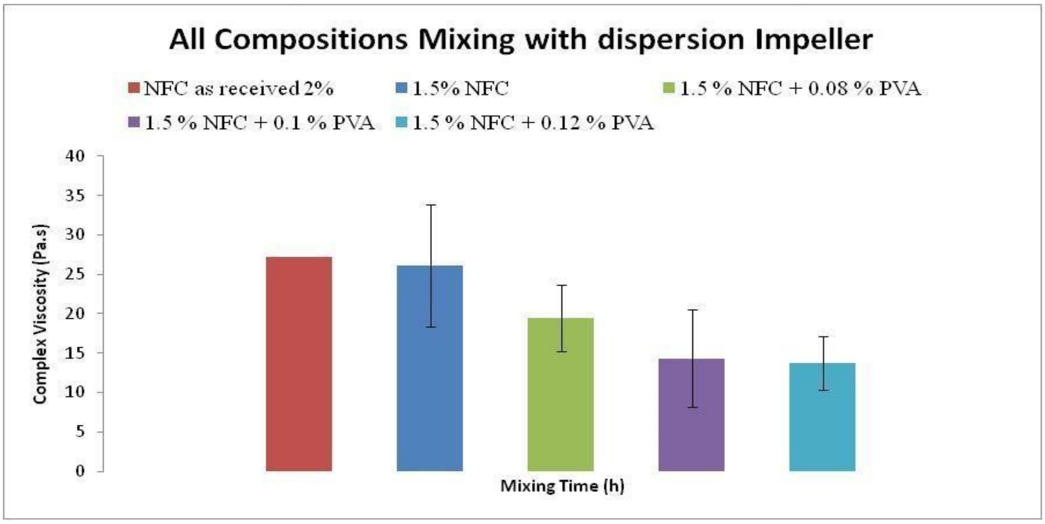

Figure 17. The complex viscosity as a function of mixing time for all compositions of PVA after mixing with a dispersion impeller for complete mixing of 48 hours

Figure 18 shows the complex viscosity as a function of mixing time for all compositions. A comparison amongst all the concentrations of PVA used in the experimental work could easily be understood from the figure. Results showed that the complex viscosity of 1.5 % NFC was higher than the NFC as received with 2%

dry solid content indicating that the beads was not effective enough to reduce the viscosity of NFC contrary to the one in which dispersion impeller was used. While mixing with PVA the viscosity of NFC had reduced appreciably at all concentrations. Out of three concentrations of PVA 0.1% reduced the viscosity most as compared to other two concentrations of PVA. It could be due to the reason that at higher concentration of PVA i.e., at 0.12% there was a reversible adsorption of PVA that detached itself from the surface of NFC and remained in the solutions that increased the viscosity of NFC. Moreover, it also provided limitation to our experimental work indicating the fact that the increased concentration of PVA is not the solution to reduce viscosity of NFC. It is the mechanism of adsorption which has to be an irreversible one where it should not detach itself from the surface of NFC.

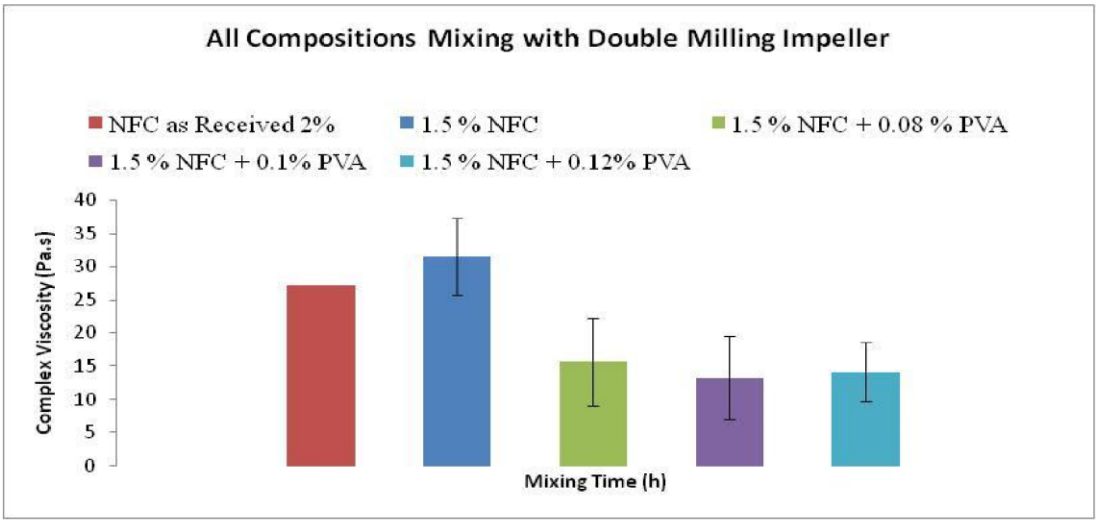

Figure 18. The complex viscosity as a function of mixing time for all compositions of PVA after mixing with a double milling impeller for complete mixing of 5 hours

Figure 19 shows the complex viscosity of centrifuged samples that were mixed both dispersion as well as double milling impeller. The centrifugation conditions were same as that of the dispersion impeller i.e., 3000 rpm, room temperature and 5 minutes time for centrifugation. Results showed that the viscosity of 1.5 % NFC when mixed with PVA using a dispersion impeller was highest as compared to all other concentrations.

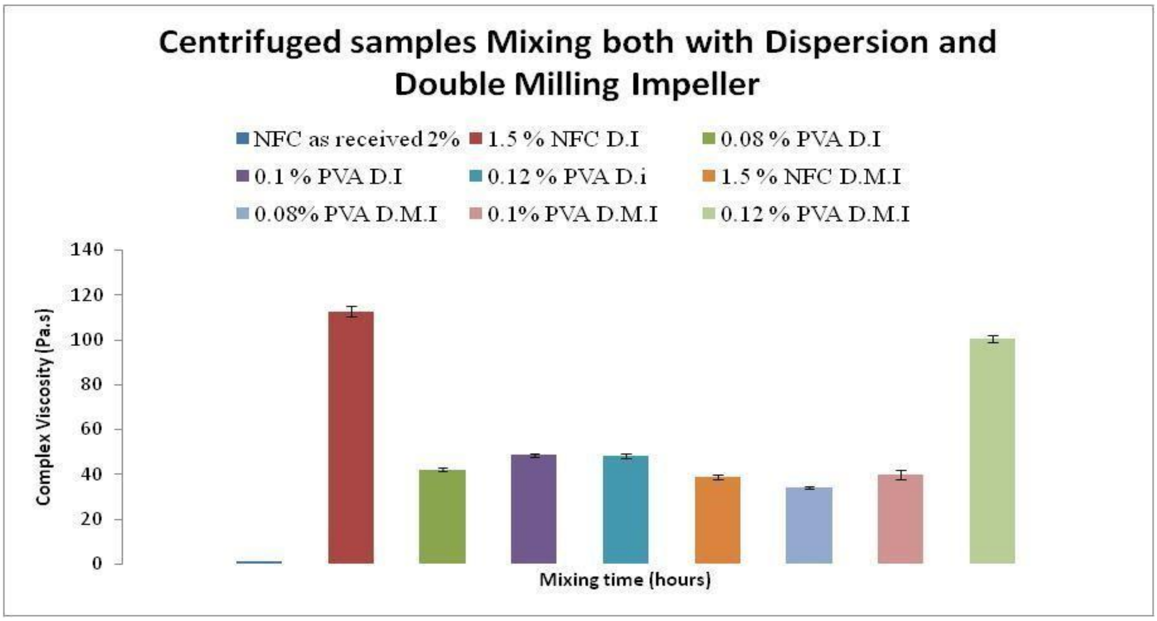

Figure 19. The complex viscosity as a function of mixing time for all centrifuged samples after mixing with both dispersion and double milling impeller for complete mixing

3.3 Dry solid content

Figures 20-23 shows the dry solid content of all compositions used in the experimental work. Dry solid content was determined every time the rheological measurement was performed. Dry solid contents were measured in order to observe if the reduction in viscosity was due to lower dry solid content or it was purely due to treatment with PVA. Results showed that despite the fact that the viscosity had reduced the dry solid content remained same for every measurement.

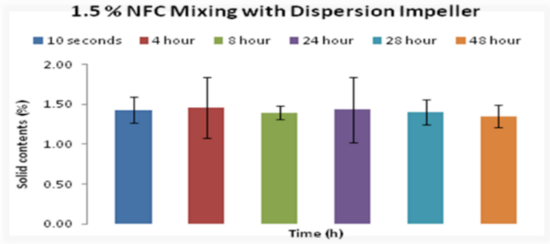

Figure 34. 1.5 % NFC Mixing With Dispersion Impeller

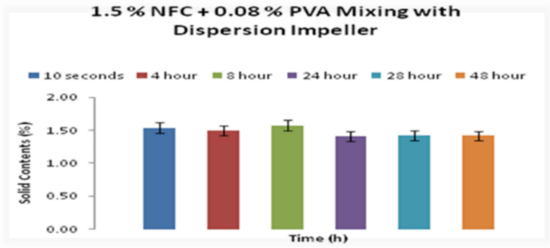

Figure 35. 1.5 % NFC + 0.08% PVA mixing with Dispersion Impeller

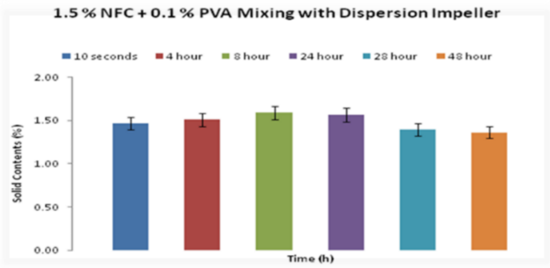

Figure 36. 1.5 % + 0.1% PVA NFC dispersion Impeller

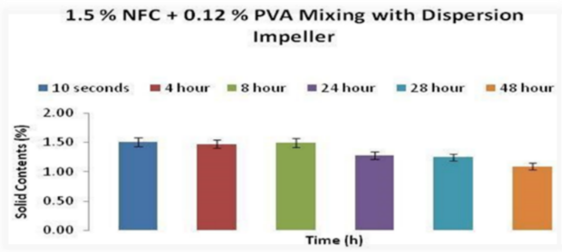

Figure 37. 1.5 % + 0.12% PVA NFC dispersion Impeller

Figure 24-27 shows the dry solid content of all compositions when mixed with double milling impeller. With different mixing technique i.e., different impeller type, rpm and mixing time results showed that the dry solid content remained same for all measurements despite the fact that the complex viscosity had decreased indicated yet again that for this mixing technique also the reduction in viscosity is purely due to the treatment of NFC with PVA. In double milling impeller beads were used for grinding the solution. Beads were isolated from the solution before subjecting to rheometer. While isolating the beads some of the solution evaporated and this could be the reason for change in solid contents.

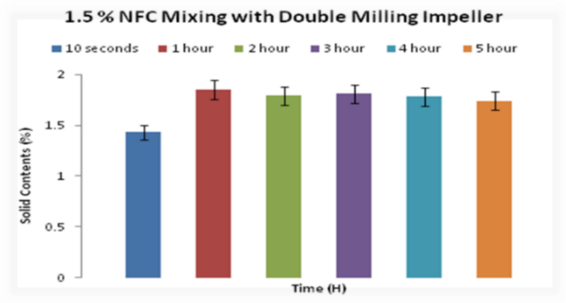

Figure 38. 1.5 % NFC double milling impeller

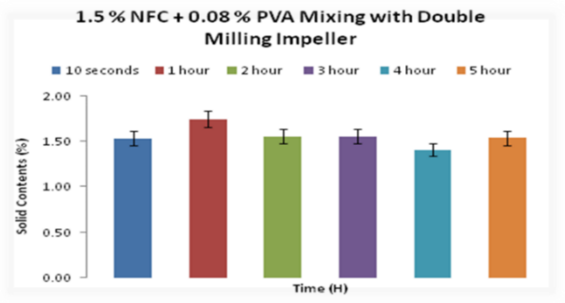

Figure 39. 1.5 % NFC + 0.08% PVA double milling impeller

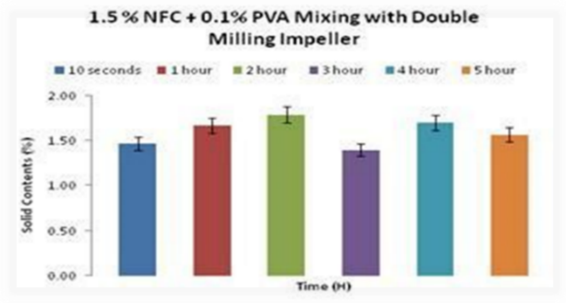

Figure 40. 1.5 % NFC + 0.1% PVA double milling impeller

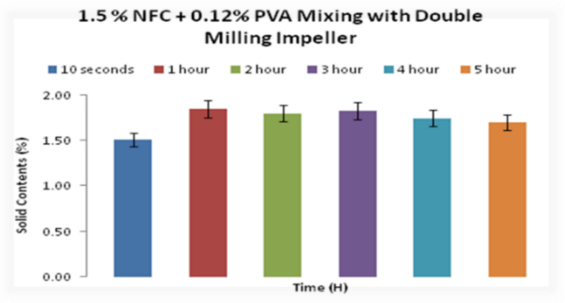

Figure 38. 1.5 % NFC + 0.12% PVA double milling impeller

Figure 28 shows the dry solid content of centrifuges samples. Centrifugation of all samples was done after treatment in a mixer. Results showed that the dry solid content of 0.12% PVA when mixed with double milling impeller is highest as compared to the other ones.

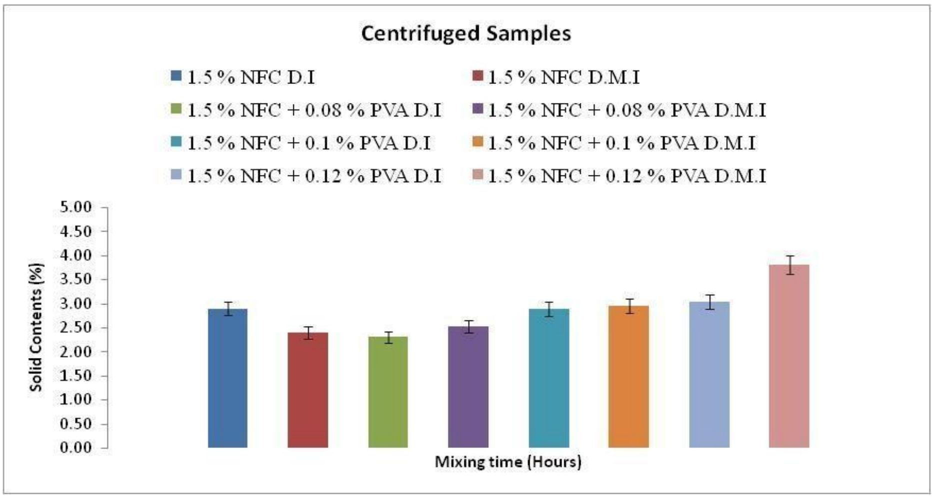

Figure 28. Dry solid content of centrifuged sample at all concentrations

3.4 Microscopic structures of all samples

Figure 29-30 shows the morphology of NFC mixed with PVA at all concentrations after mixing with Dispersion impeller. Mixing time for dispersion impeller was 48 hours. A magnification of 20X was used in order to obtain these images. These images revealed the difference in pure NFC without any PVA and that of with PVA.

Mixing with PVA showed how polymer has been helpful in making the bonds between the fibers. Without polymer addition the fibers were far apart and were randomly scattered but after adding PVA they were bonded together hence reducing the viscosity of NFC. At 0.1% PVA the viscosity was lowest. 500 pixels at the right bottom of every figure represent the length in micrometer (µm).

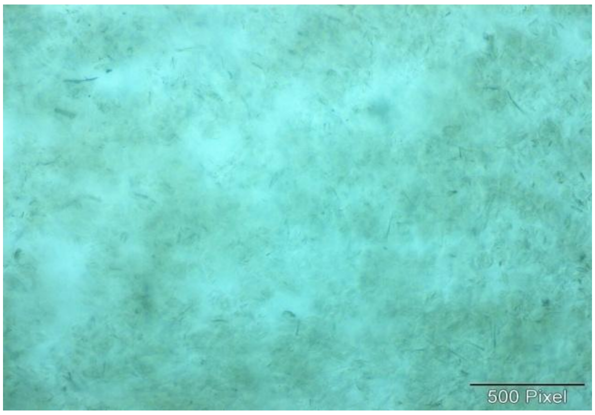

Figure 29. NFC 1.5 % + 0.12 % Dispersion impeller

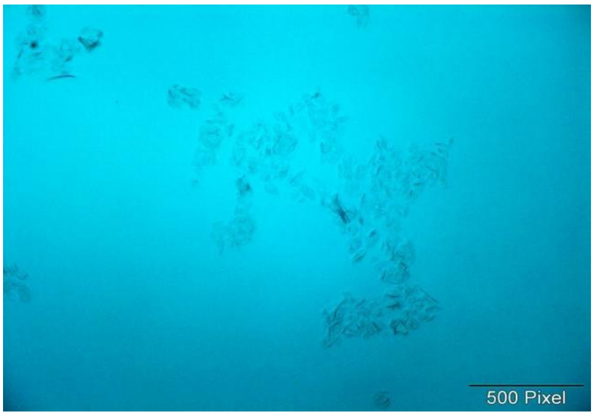

Figure 30. NFC 1.5 % + 0.1 % Dispersion impeller

Figure 31-32 shows the microscopic view of all concentrations used in the experimental work. All the images were obtained after complete mixing with a double milling impeller. All samples after being mixed with both dispersion and double milling impeller were diluted to 10% in order to obtain better results. By looking at the images it can be seen that there is an appreciable difference in the morphology of NFC when it was mixed with PVA at different concentrations. With PVA in the NFC suspension the scattered fibers were aggregated at one place hence the viscosity of NFC decreased.

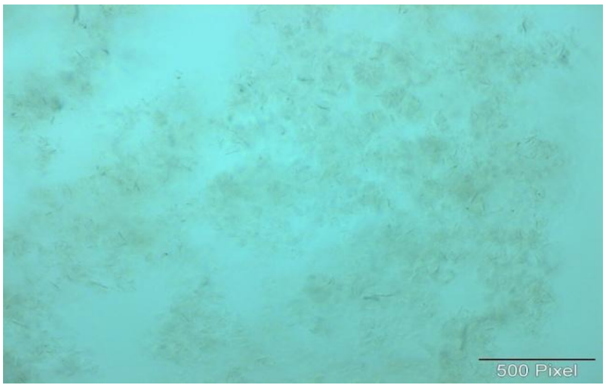

Figure 33. NFC 1.5% Double milling impeller

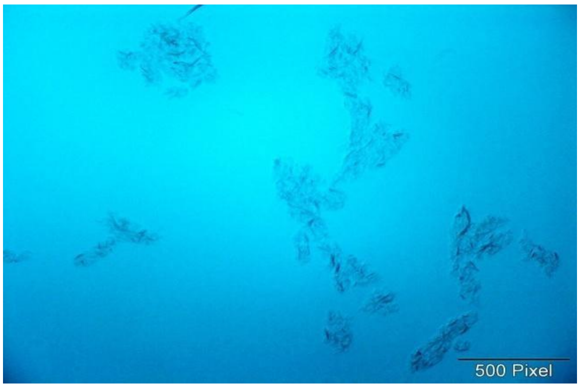

Figure 34. NFC 1.5 % + 0.08 % Double milling impeller

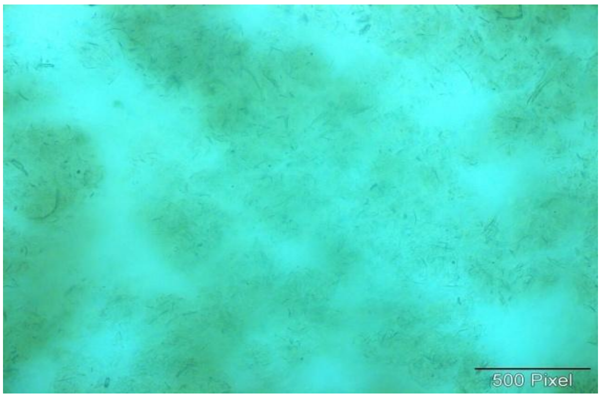

Figure 35. NFC 1.5 % + 0.12% Double milling impeller

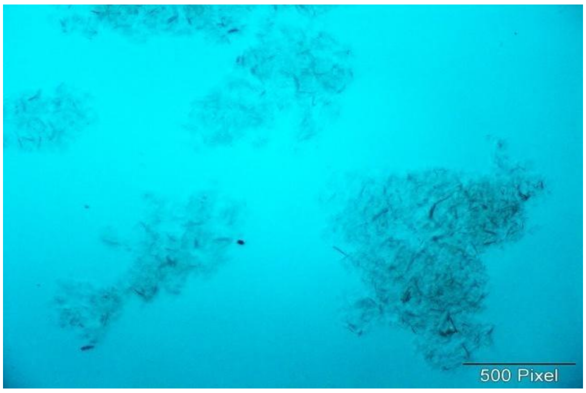

Figure 36. NFC 1.5 % + 0.1 % Double milling impeller

5. Conclusions

Form the present study it can be concluded that the viscosity of nano fibrillated cellulose was reduced by mixing it with a non-ionic polymer i.e., poly vinyl alcohol. Moreover, the viscosity was also reduced with both dispersion impeller and double milling impeller. Solid contents of the mixed samples remained constant showing the reduction in viscosity was purely due to the treatment of nano fibrillated cellulose with poly vinyl alcohol. Comparison of all three concentrations of PVA shows that an intermediate concentration of PVA i.e., at 0.1% the viscosity reduced the most. With increased mixing time more and more mechanical power was induced in to the impeller hence reducing the viscosity of the NFC. Results showed that the both the shear viscosity and complex viscosity of NFC suspension and NFC blends in double milling impeller was higher as compared to that of dispersion impeller. The type of flow when shear rate was applied on the NFC suspension as well as NFC blends (NFC+PVA) was shearing thinning.

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