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Effect of Cellulose Material-Based Additives on Dispersibility of Carbon Nanotubes

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Abstract: In nanoscience, nanotechnology is applied to various technologies, and research is actively being conducted. As the application of multi-walled carbon nanotubes (MWCNTs) in various fields increases, efforts have been made to develop dispersion and functionalization technologies. In order to effectively use MWCNT nanofluids, it is most important to solve the problem of dispersion. In this study, MWCNTs were improved in dispersibility and functionalized through various chemical and mechanical treatments. In addition, MWCNTs aggregation was alleviated by using cellulose nanocrystal (CNC) as a dispersant. The processing results of MWCNTs and CNC were analyzed through transmission electron microscopy (TEM) and the dispersion was characterized by UV–Vis spectroscopy. The addition of CNC to MWCNTs has been confirmed to have high dispersibility and improved stability compared to untreated MWCNTs, and this effect affects the quality of the machine.

Keywords: carbon nanotube; cellulose nanocrystals; dispersibility; stability; nanofluid

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1. Introduction

Nanofluids are various nanotechnology-based heat transfer fluids that are obtained by the dispersal and stable suspension of nanoparticles with a general dimension of several nanometers [1]. In general, nanoparticles have unique physical and chemical properties, compared to larger particles of the same material [2].

Carbon-based nanofluids are metals, oxides, carbides, or various carbon allotropes. Carbon particles introduce properties, such as thermal conductivity and electrical conductivity, to improve the material according to the properties of the particles [3,4]. Due to their excellent electrical and thermal conductivity properties according to the characteristics of nanoparticles, carbon nanofluids are widely used in electronic cooling systems [5], climate engineering [6], material processing [7], thermal energy storage systems [8], and heat exchangers [9]. Carbon-based nanofluids can express various properties, depending on the particle size, type, and shape. Moreover, solvent and concentration support particle properties [10]. In other words, the parameters of nanofluid efficiency are the properties of the particles, the interaction between the solvent and the carbon nanoparticles, and the method of preparation and dispersion. Carbon nanoparticles in suspension have the problem of being poorly dispersed, owing to the hydrophobic nature of the surface [11]. The dispersion of nanofluid is the indicator that determines the efficiency and performance potential of the nanofluid. In addition, dispersibility and stability degradation resulting from the aggregation of particles due to van der Waals forces cause various problems, such as reduced thermal and electrical conductivity [12]. Accordingly, various processes have been developed to increase the dispersibility of nanoparticles.

Chemical treatment modifies the particle surface through acidic solvents and improves the properties of nanofluids. It introduces functional groups to the surface and provides improved dispersibility and stability through interaction with solvents [13–15]. In addition, the procedure for mechanical treatment and addition of dispersants is also carried out. Surfactants contain hydrophobic groups and hydrophilic polar head group properties as dispersants in the particles. When cellulose is used as a dispersant, the nanoparticles are well dispersed. A stable dispersion can be achieved because the interaction occurs under the influence of oppositely charged ions of carbon particles and cellulose particles [16]. As mentioned above, the dispersion of nanofluids can be improved by mechanical and chemical treatment of particles.

Multi-walled carbon nanotubes (MWCNTs) are used in various fields and are emerging as functional materials. It is a carbon allotrope composed of carbon atoms in a hexagonal honeycomb shape, and it has distinct characteristics, such as high thermal and electrical conductivity and chemical stability [17,18]. MWCNTs can produce various nanofluids with a nano size. The scope of applications is broad, from cooling systems [19] to heating exchange fluids inside heat pipes [20] and energy storage devices [21]. However, it is not easy to simply use carbon nanotubes due to the van der Waals forces acting between them. This causes problems of poor dispersion and unstable stability [22]. To improve dispersion, Rastogi et al. [23] added a dispersion to alleviate CNT aggregation. Shen C et al. [24] exfoliated DWNTs in oleum and reacted with nitric acid. This treatment improved dispersion by decorating the DWNTs with carboxylic acid groups. Another study reported that the excessive addition of commonly used SDBS may increase structural instability. Therefore, experiments were conducted to improve dispersion by dissolving SDBS at a 100–1000 times lower concentration [25].

Therefore, in this paper, various chemical and mechanical treatments will be introduced to improve the dispersibility of MWCNT particles. Chemical treatment is used to attach functional groups to the tube and remove impurities through acid and alkaline solvents [26]. Mechanical processes seek to reduce the size of contaminants and the length of MWCNTs through ball milling [27]. However, since these processes can have a negative impact on the environment, alternative methods have been sought. This study investigates a method to alleviate van der Waals forces of MWCNTs, improve dispersibility, and stabilize them.

Rod-shaped cellulose nanocrystal (CNC) is derived from natural materials such as trees and plants [28]. Nanocrystal is structurally stable and eco-friendly so it is used for polymer composite materials [29,30]. With the above characteristics, CNC is used in mixtures to improve the properties of composites and is used to improve the dispersibility of carbon nanoparticle fluids.

In this study, an experiment was conducted to identify the effect of improving dispersibility and stability through the chemical and mechanical treatment of carbon nanoparticles and confirm the interaction with CNC materials.

2. Materials and Methods

2.1. Chemical and Mechanical Treatment of MWCNTs

Multi-walled carbon nanotubes (MWCNTs of greater than 95% purity and with less than 3% impurities with 20 nm, diameter and ~5 µm length (Carbon Nanomaterial Technology Co., Ltd., Pohang-si, Republic of Korea)) and commercial sphericity cellulose nanocrystal (CNC, Anpoly., Inc., Pohang-si, Republic of Korea) were used. Hydrochloric acid (HCl, 37%, Junsei Chemical, Tokyo, Japan), sulfuric acid (H_2SO_4, 98%, Junsei Chemical, Tokyo, Japan), nitric acid (HNO_3, 65%, Junsei Chemical, Tokyo, Japan), potassium persulfate ($K_2S_2O_8$, 99%, Duksan Pharmaceutical, Ansan-si, Republic of Korea), and sodium hydroxide (NaOH, 97%, Junsei Chemical) were used as agents for the modification of nanoparticles.

To proceed with the experiment, MWCNTs were prepared through various processes. First, a raw aspect of MWCNTs (RCNTs) was prepared. The alkalized MWCNTs (ACNTs) were obtained by a chemical reaction using potassium persulfate and sodium hydroxide. For alkalization, 1 g of MWCNTs was mixed with 100 mL distilled water (DW), and potassium persulfate was dispersed through ultrasonication for 3 h at 60 rpm. The pH reaction induces 13 levels. After the reaction, the alkalized solution was cleaned and dried in the oven for 24 h. The oxidized MWCNTs (OCNTs) were obtained through a chemical reaction using nitric acid and hydrochloric acid. The double-oxidized MWCNTs (DOCNTs) were obtained through a chemical reaction using sulfuric acid, nitric acid, and hydrochloric acid. The ground MWCNTs (GCNTs) process was obtained for MWCNTs under a wet grinding technique at a rotation speed of 500 rpm. A planetary ball milling machine (HPM-700, from Haji Engineering, Gimhae, Republic of Korea) was used for grinding process. This paper used mono-sized (3.0 mm) spherical zirconia (ZrO_2) balls for grinding, and the rotation speed was 500 rpm.

2.2. Fabrication of MWCNT-Cellulose Nanofluids

The nanofluids were used as the base in DW. In this study, five types of MWCNT-only nanofluids (RCNTs, ACNTs, OCNTs, DOCNTs, and GCNTs) were prepared at concentrations of 0.01, 0.05, and 0.1 wt%. Five types of the CNC nanofluids were prepared at concentrations of 0.01, 0.05, 0.1, 0.5, and 1 wt%. For the analysis of the characteristics of the MWCNT–CNC nanofluids, 5 types of MWCNT nanofluids with CNC were prepared for the experiments (as RCNTs–CNC, ACNTs–CNC, OCNTs–CNC, DOCNTs–CNC, and GCNTs–CNC) with concentrations of 0.01 wt% of MWCNTs mixed with 1g of cellulose. The dissolution of MWCNTs and CNC nanofluids was achieved by ultrasonication from 40 min to a maximum of 2 h. A UV–Vis spectrophotometer (X-ma-3100, Human Corporation, Seoul, Republic of Korea) was used to investigate the dispersion properties of the prepared nanofluid.

3. Structural Analysis

3.1. Morphological Observation and Analysis of CNC

To observe the structure and length of the CNC, structural analysis was performed for nanocelluloses by transmission electron microscopy (TEM). Figure 1 shows Bio-TEM images for crystal structure. Clusters containing a considerable number of individual behavior CNC are imaged in Figure 1A,B. The size of the CNC particles was confirmed to be reduced to nm by acid hydrolysis [31]. Therefore, the particles were confirmed to be nano-sized dimensional and rod shaped.

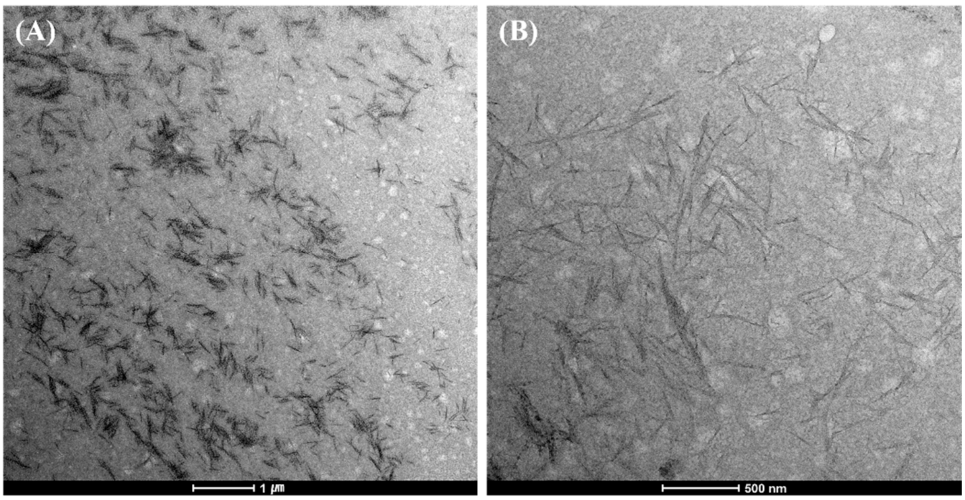

Figure 1. TEM images of CNC (**A**) 1500×, (**B**) 10,000×.

3.2. Morphological Observation and Analysis of MWCNTs

Figure 2A shows impurities through arrow of the MWCNTs. Figure 2B–E show the TEM image of purified MWCNTs in which some impurities have been removed by acid treatment alkalization. The RCNTs without treatment contained impurities, amorphous carbon, and multi-walled graphite [32]. Figure 2B shows the end of the tubes was opened and impurities were reduced on tubes outside or inside. The result for the ACNTs shows they were effective in opening the end of the MWCNTs tubes. The outer wall tubes were cleared despite mild condition, and residual graphite and inorganic compounds in the MWCNTs were removed [33]. Figure 2C shows that the treatment using nitric acid and hydrochloric acid opened the end of the tubes and removed impurities. The arrow shows that the oxidative etching with nitric acid and hydrochloric acid is well applied to the end of the tube. Treatments were not able to attach functional groups; they effectively removed impurities. Figure 2D shows the effect of the double-oxidized MWCNTs. The first purification using nitric acid and hydrochloric acid and the second oxidation using sulfuric acid and nitric acid were applied. The removal of impurities was completed, but the strong acid damaged the tubes. Figure 2E shows the tube damage through arrow of the during the ball milling process. In the ball milling process, the impurities' reduction was not noticeable. However, the reduced length of MWCNTs shows they were not entangled. Contrasting the five images confirms that the impurity and tube distinctions are effectively shown in this process.

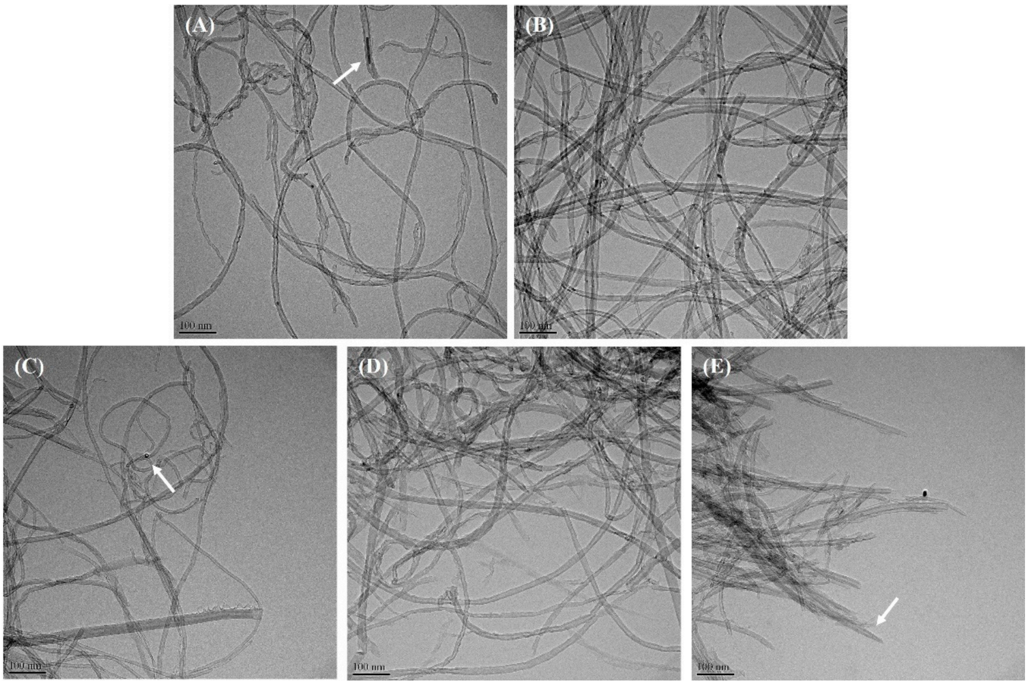

Figure 2. TEM images of the (**A**) raw MWCNTs (RCNTs), (**B**) alkalized MWCNTs (ACNTs), (**C**) oxidized MWCNTs (OCNTs), (**D**) double-oxidized MWCNTs (DOCNTs), and (**E**) ground MWCNTs (GCNTs).

3.3. Morphological Observation and Analysis of the MWCNTs–CNC

Figure 3 shows image of the nanofluid of CNC added with raw MWCNTs as TEM images. The arrow of Figure 3B shown in the image indicate the MWCNT tube between the CNC particles. The RCNT tube surface was hydrophobic, and the hydrophilic surface of the CNC mitigated the aggregation of RCNTs [34]. The result of this analysis shows the possibility that CNC and MWCNTs can be synthesized.

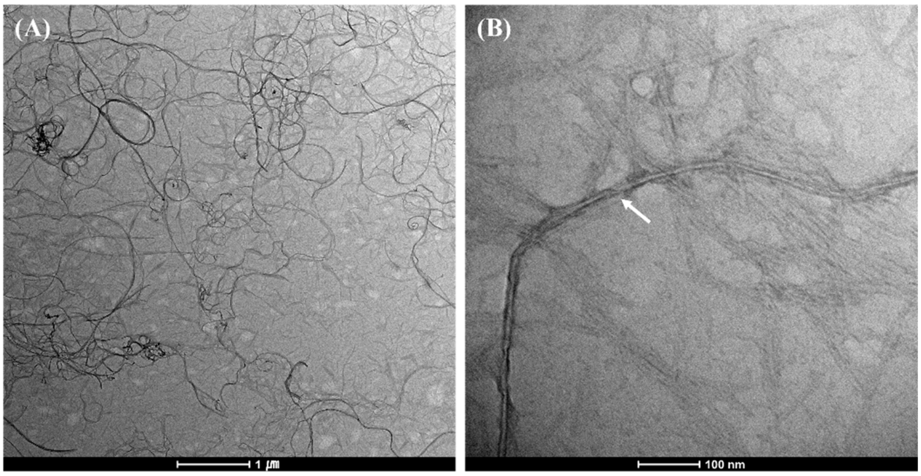

Figure 3. TEM images of RCNT-CNC with marked by arrow the raw MWCNTs (**A**) 1500×, (**B**) 20,000×.

4. Dispersion and Stability

4.1. Dispersion and Stability Characteristics of CNC

Figure 4 shows the image of the nanofluid prepared by the concentration of CNCs. CNC nanofluids were prepared at 0.01, 0.05, 0.1, 0.5, and 1 wt%, to confirm their behavior in suspension. They interacted smoothly with water and did not cause significant agglomeration over time. This phenomenon shows that the hydrophilic nature of cellulose affected the interaction with solvent, due to the abundance of hydroxyl groups in their structure [35]. Accordingly, no repulsion occurred in preparing the nanocellulose into a nanofluid.

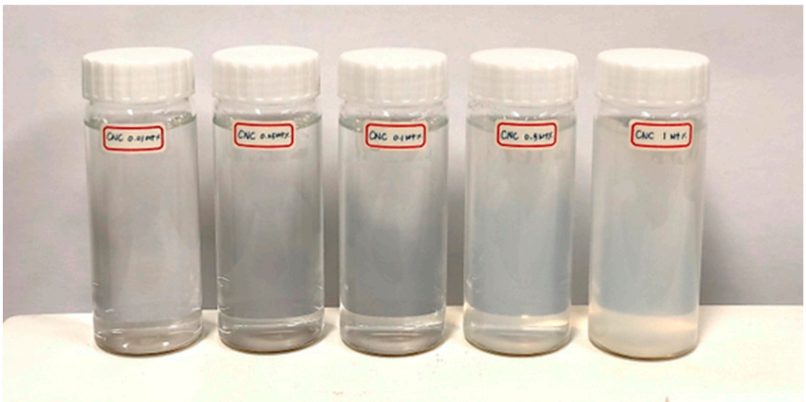

Figure 4. TEM images of the raw MWCNTs (RCNTs), alkalized MWCNTs (ACNTs), oxidized MWCNTs (OCNTs), double-oxidized MWCNTs (DOCNTs), and ground MWCNTs (GCNTs).

The dispersion characteristics of CNC nanofluids for different types of samples were investigated. After calibration with DW, CNC nanofluids were analyzed, as shown in Figure 5. Fluids show the same tendency for absorbance to increase as the concentration increase from wavelength (190 to 1100) nm. The UV–Vis spectroscopy is influenced by concentration, and color. In addition, the absorbance value varies according to the scale of the cellulose dispersed in the fluid, because the fibril scale reduces transmittance [36].

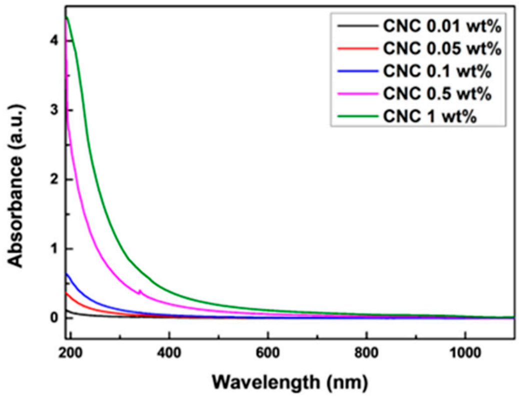

Figure 5. UV–Vis spectra measurement for the dispersion of CNC nanofluids.

4.2. Dispersion and Stability Characteristics of MWCNTs

Figure 6 shows the image of the nanofluid prepared by the concentration of all MWCNTs. All MWCNT nanofluids were prepared at 0.01, 0.05, and 0.1 wt%. Figure 6 confirms the aqua suspension of all MWCNTs through visual imagery, in which the visual images show the entanglement by van der Waals forces in each carbon nanotube. In addition, the image shows the dispersion results with and without functional groups through oxidation treatment. Additionally, in the case of ACNTs and DOCNTs, the dispersed state was maintained even after 20 days due to functional groups [37].

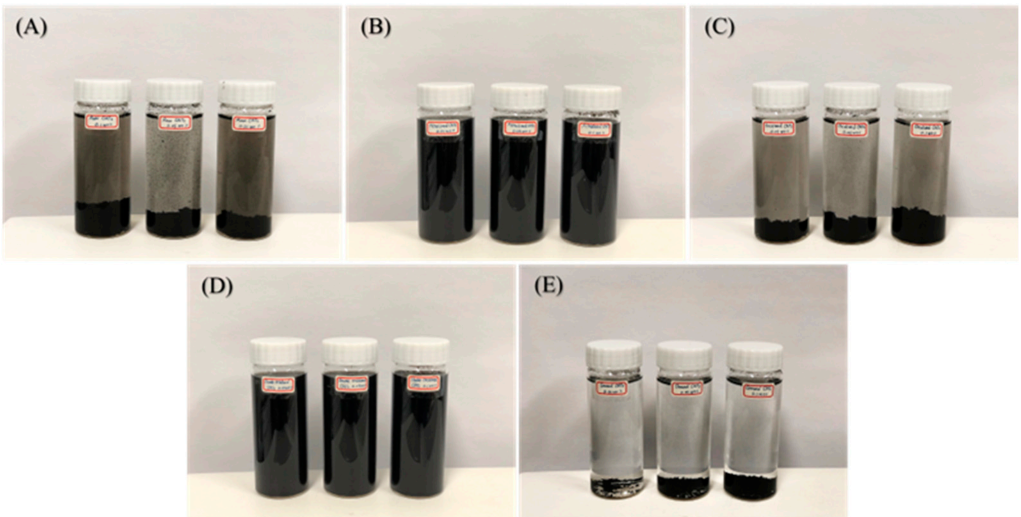

Figure 6. The images of (**A**) RCNT, (**B**) ACNT, (**C**) OCNT, (**D**) DOCNT, and (**E**) GCNT nanofluids by concentration at 0.01, 0.05, and 0.1 wt%.

Figure 7 shows the dispersion of all MWCNT nanofluids at a concentration of 0.01, 0.05, and 0.1 wt%. After calibration with DW, MWCNT nanofluids were analyzed, as shown in Figure 7. Figure 7A absorbance showed that ACNTs and DOCNTs had higher absorbance compared to untreated MWCNTs. This is considered to be an interaction among the functional groups attached to the carbon nanotubes through chemical treatment [38]. Figure 7B shows that the dispersion varies depending on the functional groups attached to the carbon nanotube, as a result of the absorbance evaluation. It was confirmed that the absorbance value of ACNTs was greater than that of DOCNTs, and as the concentration increased, the particles actively interacted. The dispersibility of the 0.05 wt% nanofluid was not easily agglomerated even when the concentration was increased, the same as at 0.01 wt%. Figure 7C shows that RCNTs, OCNTs, and GCNTs remain aggregated at

0.1 wt%. ACNTs shows the same absorbance at 0.05 wt% and 0.1 wt% even with increasing concentration. DOCNT absorbance also increases with increasing 0.1 wt% concentration.

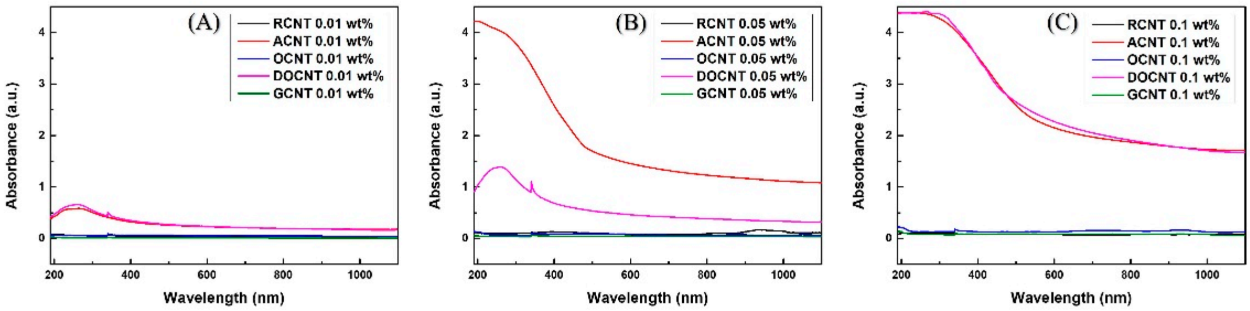

Figure 7. UV–Vis spectra measurement for the dispersion of MWCNT nanofluids by concentration at (**A**) 0.01, (**B**) 0.05, and (**C**) 0.1 wt%.

4.3. Dispersion and Stability Characteristics of MWCNTs with CNC

Nanofluids were prepared by mixing all MWCNT particles with CNC. For nanofluids analysis, nanofluids were prepared and tested at a concentration of 0.01 wt% of MWCNTs in 1 g of CNC. Figure 8A is an image before UV spectroscopy, and Figure 8B is an image confirming the nanofluid after 21 days with the naked eye. Figure 8 shows that dispersion is improved when compared to the nanofluid in Figure 6. The dispersion of RCNTs, ACNTs, and GCNTs was improved by adding CNC, but GCNTs were precipitated after 21 days.

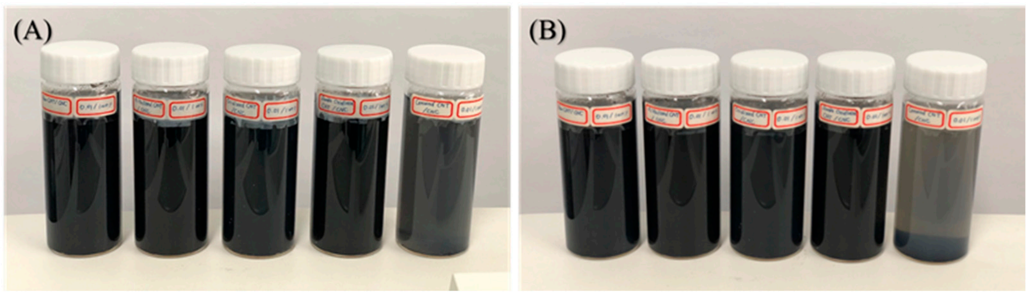

Figure 8. Photographs of the nanofluids (**A**) before 21 days (**B**) after 21 days (RCNTs–CNC, ACNTs–CNC, OCNTs–CNC, DOCNTs–CNC, and GCNTs–CNCin sequence left to right).

Figure 9 shows the dispersion of nanofluids of MWCNTs–CNC. In the absorbance evaluation, the analysis results are similar to those of the tendency of the Figure 8 images.

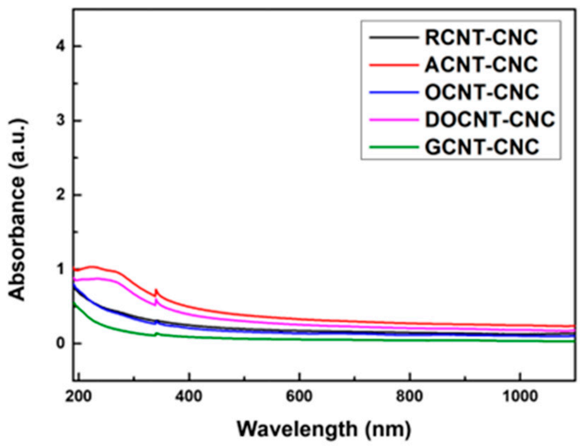

Figure 9. UV–Vis spectra measurement for the dispersion of MWCNT–CNC nanofluids.

Table 1 shows the absorbance values of carbon nanotubes–cellulose nanocrystals according to the dispersibility at 250 nm. Addition of CNC improved it by a minimum of 23% and a maximum of 95%. ACNT sand DOCNTs showed high dispersibility due to functional groups.

Table 1. Comparison of the improvement (%) in dispersion for the nanofluids from UV–Vis spectra (absorbance, at 250 nm).

Dispersion (Absorbance, at 250 nm)				
MWCNTs		**MWCNTs with CNC**		**%**
RCNTs	0.062	RCNTs–CNC	0.47	86
ACNTs	0.575	ACNTs–CNC	0.988	41
OCNTs	0.06	OCNTs–CNC	0.452	86
DOCNTs	0.653	DOCNTs–CNC	0.859	23
GCNTs	0.011	GCNTs–CNC	0.23	95

5. Conclusions

In this study, the improvement in properties of multi-walled carbon nanotubes through chemical and mechanical treatment was analyzed. Chemical treatment of MWCNTs removes metal catalysts and impurities. In addition, functional groups were attached by acid and alkaline chemical treatment. The mechanical treatment of MWCNTs did not affect the removal of impurities but was effective in reducing the length of the tube. Oxidation treatment and ball milling had the most effect on tube length. Dispersibility and stability showed high values in the order: DOCNTs > ACNTs > RCNTs > OCNTs > GCNTs. CNC interacts with tubes of MWCNTs in terms of dispersibility. The CNC continued to suspend the MWCNT nanofluids and improved the dispersibility at 250 nm, where π-bonding occurred.

In this experimental study, the basic characteristics of nanofluids were identified, and the dispersibility and stability were improved with the combination of CNC and MWCNTs. This identification is important data supporting the interaction between nanoparticles. In addition, carbon nanotubes and nanocellulose crystals are eco-friendly materials and can be applied to various eco-friendly nano technologies.

Author Contributions: Methodology, S.L.; formal analysis, S.L.; investigation A.L.; writing—original draft preparation, S.L.; material preparation: A.L.; Data collection: S.B.; writing and editing: S.L. and A.L.; review and supervision: H.J. and Y.S.; funding acquisition, H.J. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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