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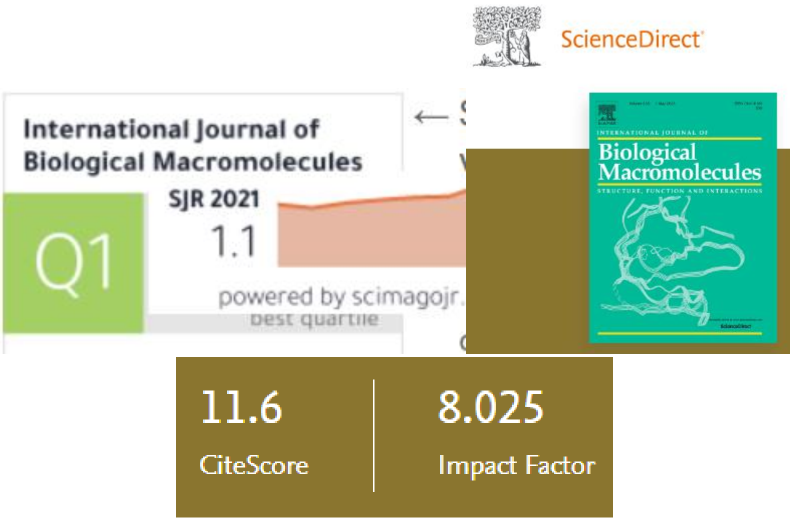
Paper 14

Synthesis and characterization of cellulose nanofibers (CNF) ramie reinforced cassava starch hybrid composites

Pengusul:
Dr. Edi Syafri, ST, M.Si



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Submission no: IJBIOMAC_2018_2328
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Corresponding author: Dr. SANJAY M R
Listed co-author(s): Dr Sudirman ., Dr Anwar Kasim, Ms Grace Tjungirai Sulungbudi, Dr Nasmi Herlina Sari, Dr edi syafri, Professor Hairul Abral

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Synthesis and Characterization of Cellulose Nanofibers (CNF) Ramie Reinforced Cassava Starch Hybrid Composites

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Synthesis and characterization of cellulose nanofibers (CNF) ramie reinforced cassava starch hybrid composites

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ABSTRACT

This study focuses on the synthesis and characterization of CNF obtained from ramie fibers reinforced with nano PCC tapioca starch hybrid composites. CNF-ramie was prepared by using chemical-ultrasonication process, while the nano-composites were made by utilizing a casting solution and glycerol as plasticizers. Physical, mechanical, and thermal properties are characterized using SEM, FTIR, XRD, TGA, and the morphology of composite samples have been analyzed through SEM. The results show that the CS/4CNF/6PCC sample has the highest tensile strength and crystallinity index of 12.84 Mpa and 30.76% respectively. The addition of CNF-ramie and PCC in nanocomposites has increased moisture absorption, crystallinity, and thermal stability properties. The SEM micrographs indicate that the CNF-ramie is bound in a matrix and the PCC is weakly bound in the tapioca starch matrix mainly due to the calcium clumps in the matrix.

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Manuscript Details

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Abstract

This study focuses on the synthesis and characterization of CNF obtained from ramie fibers reinforced with nano PCC tapioca starch hybrid composites. CNF-ramie was prepared by using chemical-ultrasonication process, while the nano-composites were made by utilizing a casting solution and glycerol as plasticizers. Physical, mechanical, and thermal properties are characterized using SEM, FTIR, XRD, TGA, and the morphology of composite samples have been analyzed through SEM. The results show that the CS/4CNF/6PCC sample has the highest tensile strength and crystallinity index of 12.84 Mpa and 30.76% respectively. The addition of CNF-ramie and PCC in nanocomposites has increased moisture absorption, crystallinity, and thermal stability properties. The SEM micrographs indicate that the CNF-ramie is bound in a matrix and the PCC is weakly bound in the tapioca starch matrix mainly due to the calcium clumps in the matrix.

Keywords	CNF-ramie; hybrid nanocomposites; PCC
Manuscript category	Carbohydrates, Natural Polyacids and Lignins

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ASSISTANT PROFESSOR
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KARNATAKA, INDIA.

Date: 13.07.2018

To,

Prof.Dr. J.F. Kennedy
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Highlights of this research work:

- This study focuses on the synthesis and characterization of CNF obtained from ramie fibers reinforced with nano PCC tapioca starch hybrid composites.
- CNF-ramie was prepared by using chemical-ultrasonication process, while the nano-composites were made by utilizing a casting solution and glycerol as plasticizers.
- Physical, mechanical, and thermal properties are characterized using SEM, FTIR, XRD, TGA, and the morphology of composite samples have been analyzed through SEM.
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- The SEM micrographs indicate that the CNF-ramie is bound in a matrix and the PCC is weakly bound in the tapioca starch matrix mainly due to the calcium clumps in the matrix.

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Abstract

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Synthesis and Characterization of Cellulose Nanofibers (CNF) Ramie Reinforced Cassava Starch Hybrid Composites

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Abstract

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Keywords: CNF-ramie; hybrid nanocomposites; PCC

1. Introduction

The use of starch and natural fibers as a material in green composites have been attracted a lot of attention due to their properties such as biodegradable, eco-friendly, low cost, abundant source [1-2]. Biodegradable starch processing can be done using several techniques such as casting solutions and injection or blow molding. Several studies of starch bioplastics have been widely studied by various resaearchers [3-5]. However, in the applicational use, this material has some deficiencies such as low permeability due to hydrophobicity, low tensile strength and low thermal stability. These weakness can be overcome by increasing the number of organic and non-organic fillers [4-5]. Cellulose nano-fibers are the new type of filler which has high efficiency and could improve both physical and mechanical properties of bioplastic based composites and has compatibility with the matrix of bioplastics [6]. The tensile strength of bioplastic could be improved along with the decreasing the hydrophobic properties using either synthesized zeolite or beidellite as the filler of biocomposite of tapioca starch [7]. The incorporation of bioplastic based starch nanofillers can also improve thermal stability, reduce moisture absorption and biodegradation [8]. Cellulose nanofiber (CNF) based biomass is an option to improve the mechanical, physical and thermal properties of bioplastic materials. The use of CNF or CNC from differentsources such as pineapple fiber, a bunch of empty palm bunches, and ramie fiber have been observed [9]. The addition of sugar palm fiber (SPF) film in a cassava starch/cassava bagasse (CS/CB) composite containing 6% CB has increased the thickness, while decreasing the density, moisture content, water solubility and water absorption. But has conversely increased relative crystallinity up to 47%, compared with 32% of CS films [10]. The tensile strength and elongation break of the bamboo nanofibers produced by casting techniques are higher than starch film at a concentration of 1.0 g/100 g [11]. Chemical modification of cellulose nanofiber (CNFs) using acetic anhydride and nanocomposites prepared by casting a solution of cornstarch with glycerol/water as a plasticizer and 10% by weight of CNF or CNF acetate (ACNF) significantly improved the mechanical properties of nanocomposites and reduced WVP and WA from TPS. In addition, the addition of nanofibers increased the rate of nanocomposite degradation of the fungus [12]. Glycerol (PS) electrolyte biocomposite (PS) with hemophytane nanocrystallite cellulose (RN) of 0-40 wt% plays an important role in strengthening composites. Tensile strength and Young's modulus increase from 2.8 MPa to 6.9 MPa for PS films and from 56 MPa to 480 MPa for PS films with increasing RN content from 0 to 40% by weight [13]. From the previous research works it is observed that the synthesis and characterization of cellulose nanofiber from ramie fiber reinforced calcined calcium carbonate have not been investigated.

Therefore, this study is carried out to investigate on the synthesis and characterization of CNF from ramie fiber which reinforced by hybrid nanocomposite PCC (precipitated calcium carbonate) with tapioca starch matrix. The synthesis of CNF has been processed using a chemical and ultrasonication method. The chemical method involves pulping, bleaching and hydrolysis process using sulfuric acid, while the ultrasonic process has been conducted to generate CNF-ramie and tapioca starchas a matrix and PCC. Nanocomposite was characterized using SEM, Universal Testing Machine (UTM), XRD, FTIR, and TGA.

2 . Material and Methods

2.1 Materials

Ramie fiber is obtained from the main stem by removing the bark (fertilizing) from a Ramie plant belonging to the family of Uritcaceae. Pure chemicals such as sodium hydroxide (NaOH) of 98%, potassium hydroxide (KOH), sodium chloride ($NaClO_2$), acetic acid (CH_3COOH), sulfuric acid (H_2SO_4), glycerol, $CaCO_3$/PCC and starch tapioca have been used for fiber purification.

2.2 Preparation of ramie cellulose nanofibers

Figure 1 & 2 shows the CNF ramie insulation process. The ramie fiber is cut in the range of 10-20 mm and dried naturally in the sunlight for 3 days so that its water content reaches 9-10% range. There are four steps to obtain CNF-ramie, as follows: First, pulping process where they have immersed fibers in 18% NaOH solution for 2 hr at a temperature of 170^{o}C at a pressure of 7-9 kg/cm^2 [14]. Next fiber in the form of the pulp was washed with free alkali followed by a bleaching process using a mixture of sodium chloride ($NaClO_2$) and Acetic Acid maintained in a solution of pH = 5 at 70 °C for 2 hours and then washed with mineral water until the neutral acidity approximates pH = 7. In the second step, the fibers are bleached using 4% KOH at 80^0 C for one hour to reduce non-cellulose content [15]and washed with mineral water so as to achieve pH = 7. Next in the third step, the fibers are rebleached in 5% $NaClO_2$ solution and acetic acid with pH = 5 at 70^0 C for one hour and then washed with mineral water until pH = 7. In the last fourth step, the hydrolysis process was processed by 30% H_2SO_4 for one hour with a fiber concentration ratio and solution of 1: 8.75 [16]. Finally, the fiber suspension of the hydrolysis process of sulfuric acid was carried out by ultrasonication at 70 ^{0}C using a sonicator (Ultrasonic 750W) [17] with different times of 1, 1.5 and 2 hrs to obtain cellulose nanofiber (CNF)-ramie.

2.3 Production of CNF-ramie/PCC hybrid composite

Production of CNF-ramie/PCC hybrid composite as shown by Syafri et al.[9]. Cassava starch dissolved in distilled water (5% v/v) and CNF (% b/b) from cassava starch/tapioca starch with differentconcentrations of mineral water (see in Table 1).

Glycerol (30% w/w tapioca starch) is added to the mixture as a plasticizer. Then, the tapioca/CNF-rami/glycerol starch mixture was heated to 65-75 ^{o}C at 350 rpm to gelatinization and poured to the mold dimension of 11 x 9.5 x 0.3 cm^3. The mold put on the ultrasonic bath for 15 min to remove air bubble and dried in the oven on temperature 37 ^{o}C for 17 hr. Bioplastic of hybrid composite CS/CNF/PCC stored in the desiccator for 24 hr before characterizing test.

2.4 Morphology of CNF-ramie and hybrid nanocomposites

The surface of a tested sample of CNF-ramie was observed by using TEM (Model: Tecnai G^2 20 S-TWIN) on 200 kV for microstructure characterization of ramie fiber suspension. The ultrasonication process for 120 min, treated ramie-fiber dropped to Holley Carbon Grid and dried at room temperature for 4 hr.)

Furthermore, the surface morphology of hybrid nanocomposites was observed by using SEM (Model: JSM 6510 from JEOL) with voltage and current of 16 kV and 8 mA respectively. The test sample put on the stub sample of SEM. Then, samples were gold-sputtered for 5 min to a thickness of approximately 10 nmto impart conductivity. SEM images were collected at different magnifications to assureclear images.

2.5 Tensile Strength

The tensile strength of nanocomposite samples were tested on Universal Testing Machine (Model: Strograph-R1, Shimadzu). at room temperature 25^{o}C by following standard of ASTM D638 [18], which has crosshead speed 2 mm/min with load cell 5 kN. A total of 7 samples were tested foreach parameter, and the average standard deviation values were reported.

2.6 X-rayDiffraction (XRD)

The nanocomposites samples were analyzed by XRD (Model: Empyrean X-ray Diffraction System from Malvern Panalytical) using diffractometer circuit of analytical. The radiation of CuKα recorded on wave number 1.54060 with voltage and current on 40 kV and 30 mA respectively. The step size was 0.02°, the step scan was 10.16 s, and the 2θrange was 10°–90°. The integrated intensities of the Bragg peaks in the spectra of the nanocomposite samples were calculated, and the crystallinity was evaluated based on the percentage crystalline intensity (I_C). The crystallinity index (CI) was calculated using Equation 1 [19]:

$$CI = \frac{I_c - I_{am}}{I_c} \times 100\% \quad \text{...(1)}$$

Ic is the crystalline intensity and I_{am} is the amorphous intensity.

2.7 Moisture Absorption

The sample of hybrid nanocomposite of dimision 2x1 cm^2 wete dried in the oven at temperature 60^oC. Sample stored in the plastic container with humidity 78±2% and weighed every 30 min for period process 210 min [20]. They stored at ambient temperature for 18 hr to get precise value by using precision scales 0.1 mg. The absorption of water calculated by using equation 2 [21]:

$$MA = \frac{Wh - W0}{W0} \quad \text{..(2)}$$

MA is moisture absorption; W_h is final weight; W_0 is initial weight.

2.8 Fourier Transformed Infrared Spectroscopy (FTIR)

Perkin Elmer Spectrum Fourier transforms infrared spectrometer was used to derive the FTIR spectra of the nanocomposites samples in KBr matrix with a scan rate of 32 scans per minute at a resolution of 2 cm^{-1} in the wave number region between 400 cm^{-1} and 4000 cm^{-1}. Over-fills in the detector due to the divergence of the beam may end up with cumulative transmittance percentage of 100 ± 15%. The chopped fiber samples were groupadded to a fine powder using a mortar and pestle and then mixed with KBr powder. They were then pelletized by applying pressure to prepare the specimen to record the FTIR spectra. The presence of free functional groups in nanocomposites is determined by FTIR.

2.9 Thermogravimetric analysis (TGA)

The thermal stability behavior of the nanocomposite samples was assessed by TGA (Model: Perkin Elmer-TGA 4000). To avoid oxidation effects, the TGA analysis were carried out in a nitrogen atmosphere at a flow rate of 40 ml/min. Ten milligrams of nanocomposite sample was crushed and kept in an alumina crucible to avoid the temperature variations measured by the thermocouple. The heating rate is maintained at 10°C/min for heating it from 50-400°C with a flow rate of 10°C.

3. Result and discussion

3.1 Physical properties and morphology of CNF-ramie fibers

Figure 3a shows that the diameter of cellulose-ramie decreased with the increasing of ultrasonic time due to the particle size distribution of cellulose-ramie decreased. The highest value of diameter CNF fiber approached on the ultrasonic time of 60 min instead of either 90 or 120 min of 193, 67.11 and 34.32 nm respectively. The result was confirmed with TEM images (see in Figure 4a). Figure 4b shown that formed crystal structure is hexagonal (002). Bothchemical and ultrasonic treatment which implemented to ramie fiber generated uniformly particle size of CNF-ramie 99.8%. It is due to the diameter of CNF-ramie which produced by using chemical-ultrasonication method is smaller than which of using strong acid hydrolysis method conducted by Lu et al. Its particle size of ramie is about 85.4 nm [22]. On the other hand, Teixeira et al [3] produced cellulose nanofiber with diameter 1-11 mm and length 360-1700 nm from tapioca starch using strong acid hydrolysis. The acid treatment with high concentration will affect the disintegrated fiber [23].

The alkaline treatment of ramie fiber with operating temperature 170 ^{0}C affected hemicellulose to be hydrolyzed and dissolved in the water. Bleaching treatment conducted to remove the lignin content. This fact due to oxidized lignin by chlorine so it will degrade and create hydroxyl, carbonyl, and carboxylates group; thus easily soluble in alkali medium [23]. Cellulose nanofiber obtained due to loss of hemicellulosic and lignin bond on crude fiber. Mechanical treatment conducted to reduce the particle size of fiber either diameter or length and separate the bunch of cellulose fiber [24].

3.2 Physical properties and morphology of CNF-ramie /PCC hybrid nanocomposites

The surface of hybrid nanocomposite CS/CNF/PCC can be seen in Figure 5. Its shape looked uniform and not porous on the surface. On some variations of the sample, whiteness agglomerates appeared. It is due to PCC agglomerate in the tapioca starch matrix. Visually, some spot of agglomerate PCC caused by immiscible blend between PCC and its matrices. It confirmed with the result of XRD. There is a peak of PCC on $2\theta=29.6^{o}$C and another different angle of 2θ. CNF-ramie on the nano-composite cannot be seen due to nano-sized particle while

SEM detected on the micron scale.

3.3 Tensile strength and strain CNF-ramie/PCC nanocomposites hybrid

CNF-ramie implemented on the filler of bio-plastic based tapioca starch. It purposed to improve the mechanical properties of bio-plastic. The tensile strength and elongation at break of hybrid nano-composite CNF/PCC with various concentrate showed in Figure 6 and 7. The tensile strength of nanocomposite based tapioca starch increased with the decreasing of PCC content in the matrix. This fact due to an immiscible blend of PCC and tapioca starch, there are many entrapped air bubble in the matrices of nano-composite. Edhirej et al [25] stated that the similar result, the result of tensile strength is depending on the tapioca starch content.

Furthermore, the mechanical properties of tested sample 6CNF/4PCC decreased due to aggregation of CNF and PCC on matrices of nano-composite, thus it cannot hold the load evenly. This fact is similar to other study composites based corn starch reinforced by nano-sized of $CaCO_3$. Its tensile strength decreased with the increasing of filler content [2].

The highest value of tensile strength on tested sample 4CNF/6PCC is about 12.84 MPa compared with pure CS 2.51 MPa as a control sample. The previous study stated that the addition of $CaCO_3$ as much 0.06% improved the tensile strength from 1.4 MPa (without $CaCO_3$) to 2.42 MPa (with $CaCO_3$) in the bioplastic based corn starch[2]. Syafri [5] reported that the addition of PCC as much 4% improved the tensile strength from 1.65 (without tapioca starch) to 2.60 MPa (with tapioca starch) in the matrix of bio-plastic based tapioca starch. Moreover, Lu [22] reported that the addition of cellulose nanofiber-ramie improved both tensile strength and young modulus significantly from 2.8 MPa and 55.9 MPa to 6.9 MPa and 479.8 MPa. On the other hand, the elongation at break decreased significantly from 94.2% to 13.6%.

Figure 6 showed that the tensile stress decreased from 12.84 to 5.78, 7.46 and 11.23 MPa with the increasing of CNF content 6CNF/4PCC, 8CNF/2PCC,and 10CNF/0PCC respectively. This is due to the increase of insoluble material content with the matrix which already reported with a previous study [26-27].

The addition of CNF-ramie and PCC could reduce the elongation of hybrid nano-composite significantly. The elongation at break reduced significantly from 49.55% to 3.69, 3.42 and 2.05% with the increasing of CNF/PCC content as much 0CNF/10PCC, 2CNF/8PCC,and 4CNF/6PCC. Nevertheless, the addition of CNF-ramie along with the reduction of PCC could improve the elongation at break. This occurs on 6CNF/4PCC, 8CNF/2PCC,and 10CNF/0PCC. Their elongation is increased to be 18.95, 5.23 and 4.58% respectively.This is due to the diffusion of CNF-ramie and PCC in the matrices of nanocomposites. Hybrid nano-composite contained 4% of CNF and 6% of PCC had the best of tensile strength. The result showed that the tensile strength had reversely resulted in elongation properties. This is proved by a previous study [28].

3.4 XRD analysis

Figure 8 shows that diffraction of XRD from the raw material of hybrid nano-composite. Figure 9 shows that diffraction of XRD from mixed hybrid nano-composite and several various filler CNF-ramie and PCC. Hybrid nanocomposite had a similar pattern and showed crystalline peak after adding CNF and PCC content.The diffraction pattern of nanocomposite based tapioca starch with filler CNF-ramie/PCC on angle $2\theta=5\text{-}40^{o}$ shows that improvement of degree crystallinity of the material. Crystallinity index of bio-plastic composite (Table 2).

Figure 9 shows that the X-ray diffraction of pure bioplastic as a control had low intensity, narrow diffracted peak, and low degree crystallinity. The addition of CNF-ramie will improve the crystallinity of hybrid nanocomposite, so that improve the tensile strength of the material. The highest value of crystal index could obtain by the addition of CS/4CNF/6PCC. This is due to the immiscibleblend of PCC with cassava starch matrix. This is showed from a crystalline peak of PCC on angle $2\theta=23.13$, 29.48, 36.00 and 39.52^{o}. The addition of PCC content improved the crystallinity of hybrid nanocomposite. The lowest value of crystal index is on nano-composite CS/0CNF/10PCC. The previous study also stated that the crystallinity of nano-composite based bio-plastic increased with the increasing of fiber content [29].

3.5 Moisture absorption analysis

Figure 10 shows that the moisture absorption of the sample with various variations.Initially, the sample has had a high capacity for moisture absorption. However, the acceleration of moisture absorption of sample gradually decreased after 3.5 hr. The average of moisture absorption on 3.5 hr for CS (control), CS/0CNF/10PCC, CA/2CNF/8PCC, CS/4CNF/6PCC, CS/6CNF/4PCC, CS/8CNF/2PCC and CS/10CNF/0PCC is 23.12, 18.95, 22.09, 22.86, 20.89, 23.26 and 25.13 respectively.

Figure 10 shows that the resistance of sample moisture absorption increased with the increasing of PCC content. The highest value of resistance of sample moisture absorption is on mixture CS/0CNF/10PCC. This is due to PCC is hydrophobic inorganic compound, its hydrophilic properties are worst than cassava starch and CNF-ramie. While the sample of CS is the easiest sample to absorb water due to its natural properties hydrophilic and probably microporosity of bio-plastic. When the addition of other-type of filler in the hybrid nano-composite could improve the interface bond between the matrices, fiber,and filler of PCC simultaneously. Meanwhile, the homogeneity of the sample could resist the water molecule to diffuse to the matrices[4].

3.6 FTIR analysis

Figure 11 shows that the spectrum FTIR for the characterized samples such as CS (control), CS/0CNF/10PCC, CS/2CNF/8PCC, CS/4CNF/6PCC, CS/6CNF/4PCC, CS/8CNF/2PCC,and CS/10CNF/0PCC. At the absorbance on range 3200-3800 cm^{-1} is the peak of stretching O-H.The FTIR spectrum of both starch and fiber is similar due to its similar main structure cellulose. The spectrum of O-H groups in starch detected on range 3200-3800 cm^{-1} showing that the high hydrophilic content of starch [30]. On the other hand, the FTIR spectrum of bio-plastics based tapioca starch as control which has absorption bands of stretching O-H and C-H are on 3429 cm^{-1} and 2926 cm^{-1} respectively. The absorption bands of 1627 cm^{-1}attributed to the scissoring of O-H and bonding water molecules. The stretching C-O from C-O-C in the aryl-alkyl-ether groups at 1336 cm^{-1}. Moreover, the stretching of C-O from C-O-C in the ring of anhydroglucose detected on the FTIR spectrum of 1029 cm^{-1}[31]. At the spectrum of range 900-1030 cm^{-1} related to the stretching of anhydroglucose [32].At the band,1500-1600 cm^{-1} detected due to the diversion of water in starch [33].

3.7 TGA analysis

The thermal analysis of hybrid nanocomposite CNF-ramie/PCC is shown in Figure 12. The different concentrate of CNF-ramie and PCC detected by using mass degradation method. Table 3 shows that the temperature degradation and loss mass sample of the hybrid nanocomposite. In the first stage, the loss mass sample on temperature below 100^{o}C associated with water loss, while loss mass sample on temperature range 100-225^{o}C associated with water evaporation and also plasticizer. In the second stage, the loss mass sample on range temperature 225-350^{o}C which had peaked on 330^{o}C associated with ether and unsaturated structure which formed in thermal condensation of hydroxyl groups of starch chains duringremoval water and other molecules. Further, the final stage, it is on range temperature 350-500^{o}C caused by disintegrated residue which generated from oxidative atmosphere process. [34]

Figure 11 shows that the loss mass sample and decomposition on initial temperature due to the different concentrations of CNF-ramie/PCC. The loss mass sample on temperature 500 ^{o}C decreased with the increasing of filler content. The lowest value of mass residue in bio-composite 10% is on mixture 10C/0PCC. On the other hand, the highest value of mass residue in bio-composite 20.1% is on mixture 2CNF/8PCC. While control (CS) had loss mass sample of 19%. The mass residue of hybrid nanocomposite for various sample 0CNF/10PCC, 2CNF/8PCC, 4CNF/6PCC, 6CNF/4PCC, 8CNF/2PCC and 10CNF/0PCC is 11.6, 20.1, 14.9, 10.8, 14.3 an 10% respectively. These results are similar to Prachayawarakorn et al. [31].They stated that the thermal stability is increased with the increasing of filler content due to a good adhesion effect between fiber and matrices. Finally, it could reduce the loss mass sample.

Conclusion

Chemistry-ultrasonication is used to isolate cellulose nanofiber ramie. Hybrid compound strengths reinforced by CNF-ramie / PCC were found to be lower than CNF variations (0-10 wt%) and PCC (10-0 wt%). The highest value of nano-composite hybrid tensile strength of CNF-rami / PCC 12,84 MPa was obtained on CS/4CNF/6PCC mixture with crystal index of 30.76%. SEM morphology shows that there is an interaction between matrix and CNF-rami / PCC filler due to homogeneous nano-filler dispersion, but the PCC distribution shows heterogeneous. The value of mechanical strength and thermal stability is significantly increased from the CNF-rami/CNC hybrid nano-composite.

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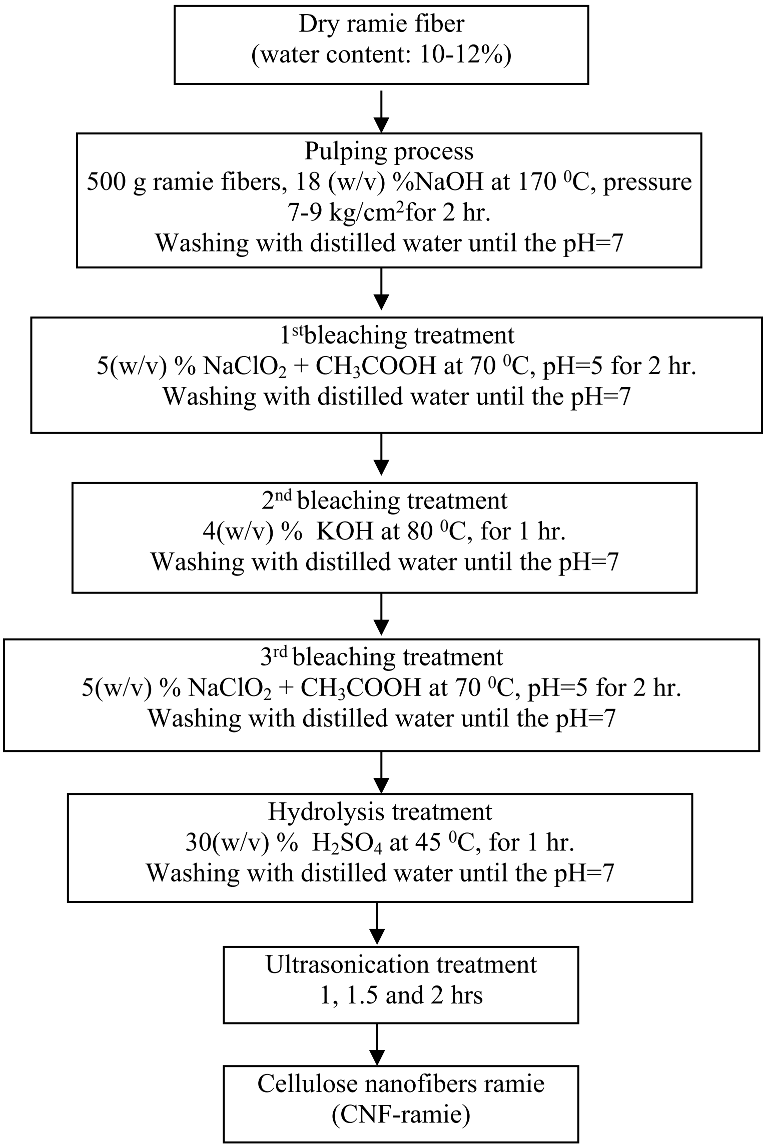


Figure 1. Steps involved in synthesis of cellulose nanofibers ramie

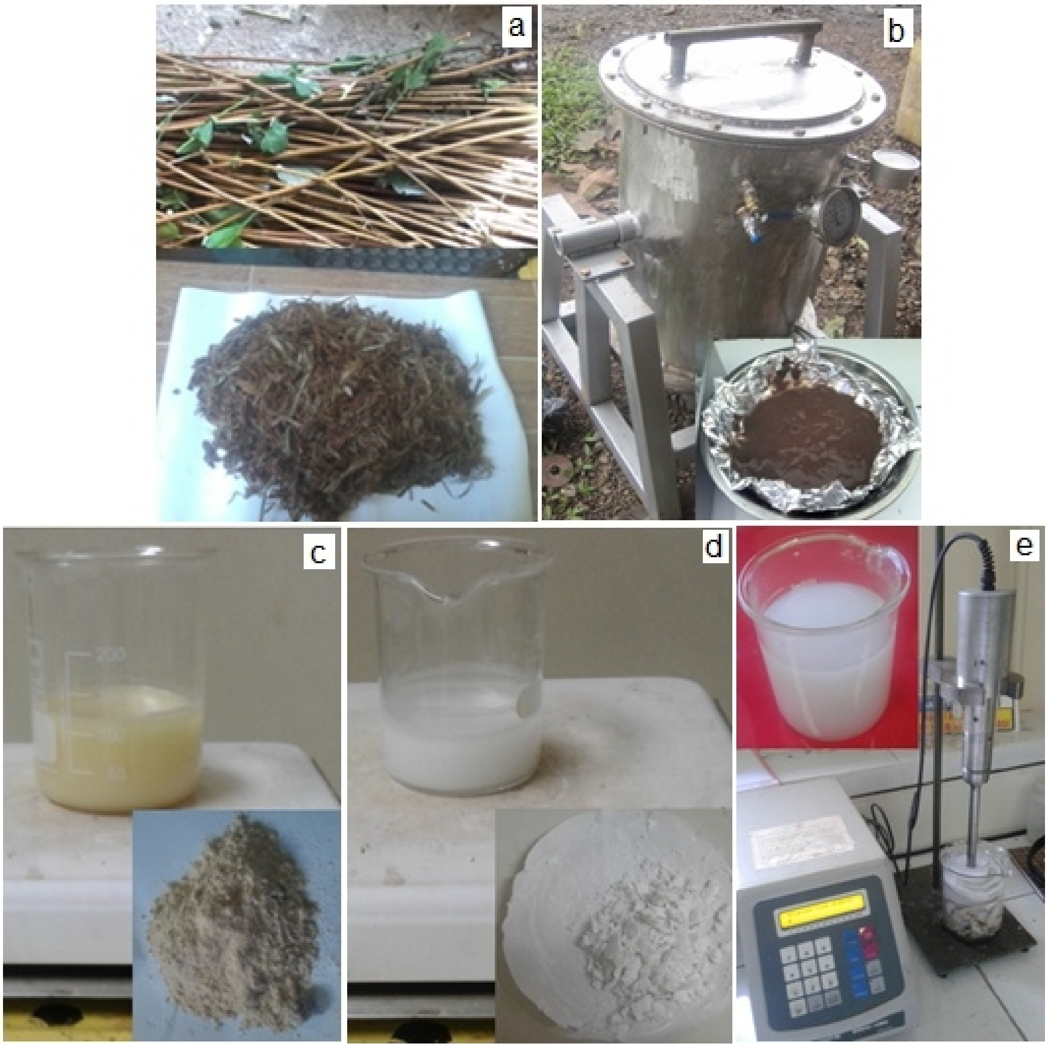
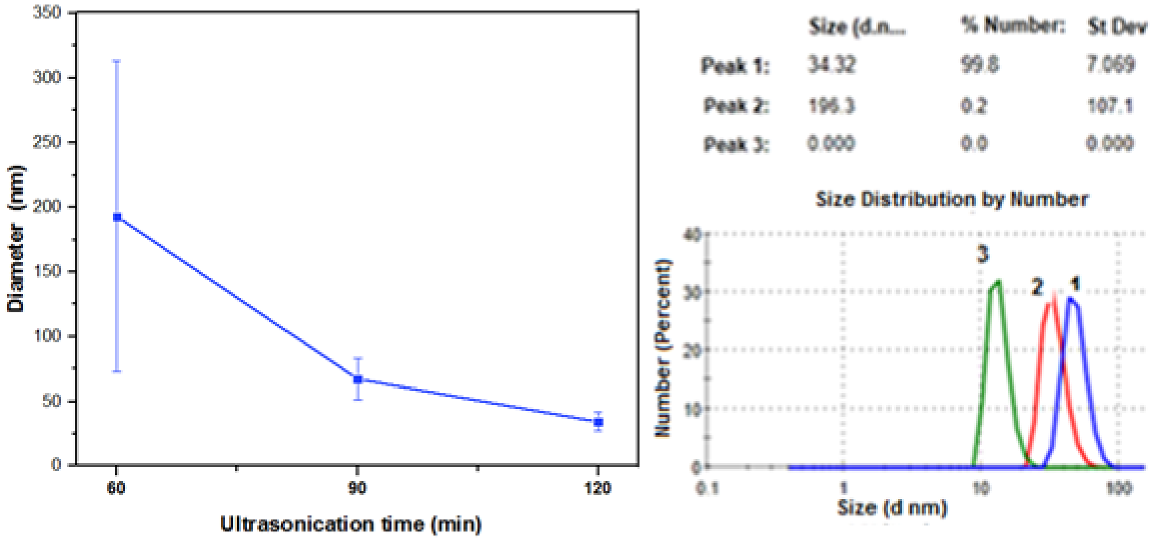
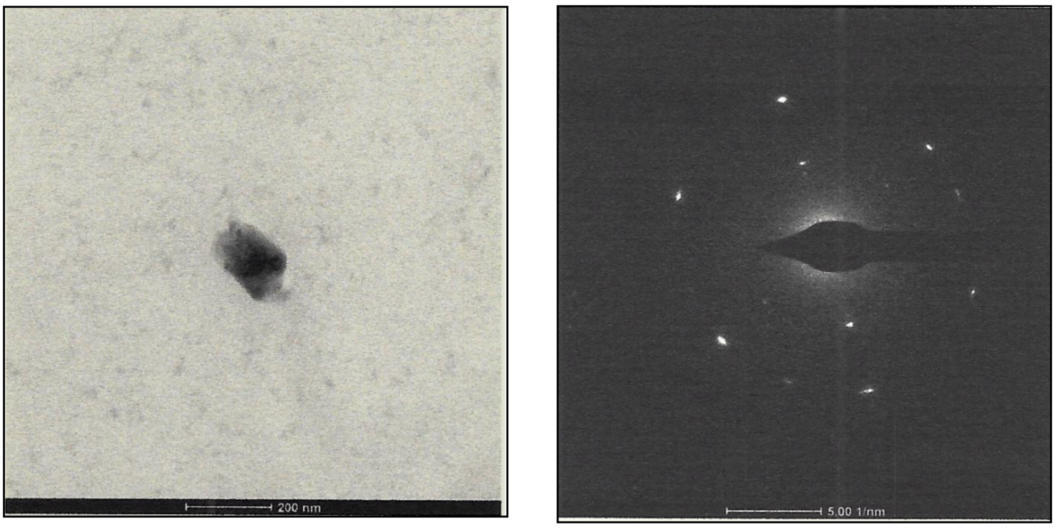


Figure 2. Synthesis of CNF-ramie process, a) raw, b) pulping ramie, c)Bleaching ramie, d)hydrolysis ramie, e) ultrasonication ramie process



a b

Fig. 3. a) Effect of ultrasonication time vs diameter of CNF, b) Size distribution of CNF PSA test results 2 hr.



a b

Fig. 4. TEM Photographs PSA for 2 hours of ultrasonic treatment, a) CNF-ramie on 200 nm scale, b) Selected Area Electron Diffraction (SAED) CNF-ramie on 5.00 1/nm scale.

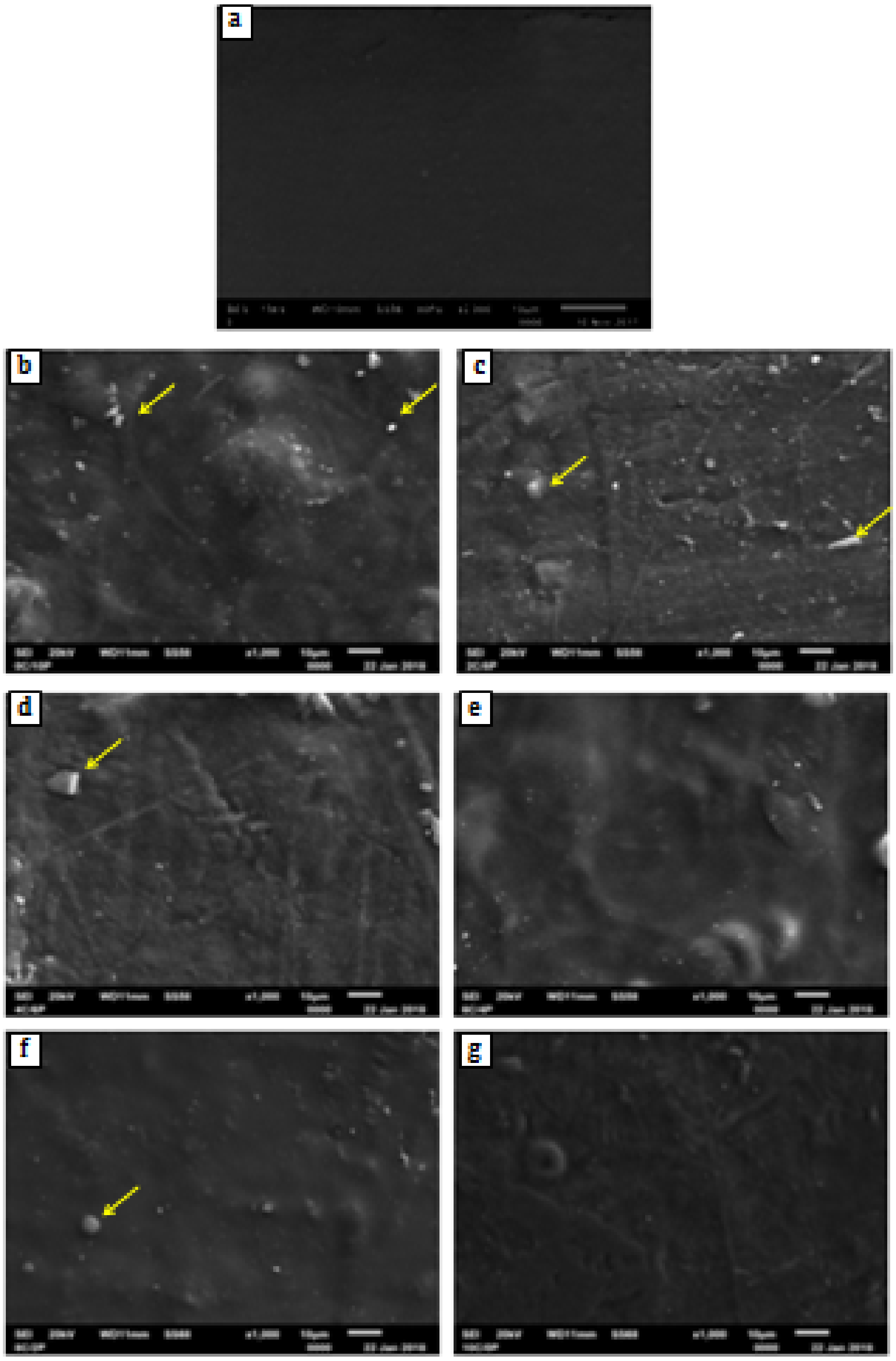


Figure 5. SEM imagesof CNF-rami/PCC hybrid nanocomposites, a)CS (Control), b) CS/0CNF/10PCC, c) CS/2CNF/8PCC, d) CS/4CNF/6PCC, e) CS/6CNF/4PCC, f) CS/8CNF/2PCC and g) CS/10CNF/0PCC.

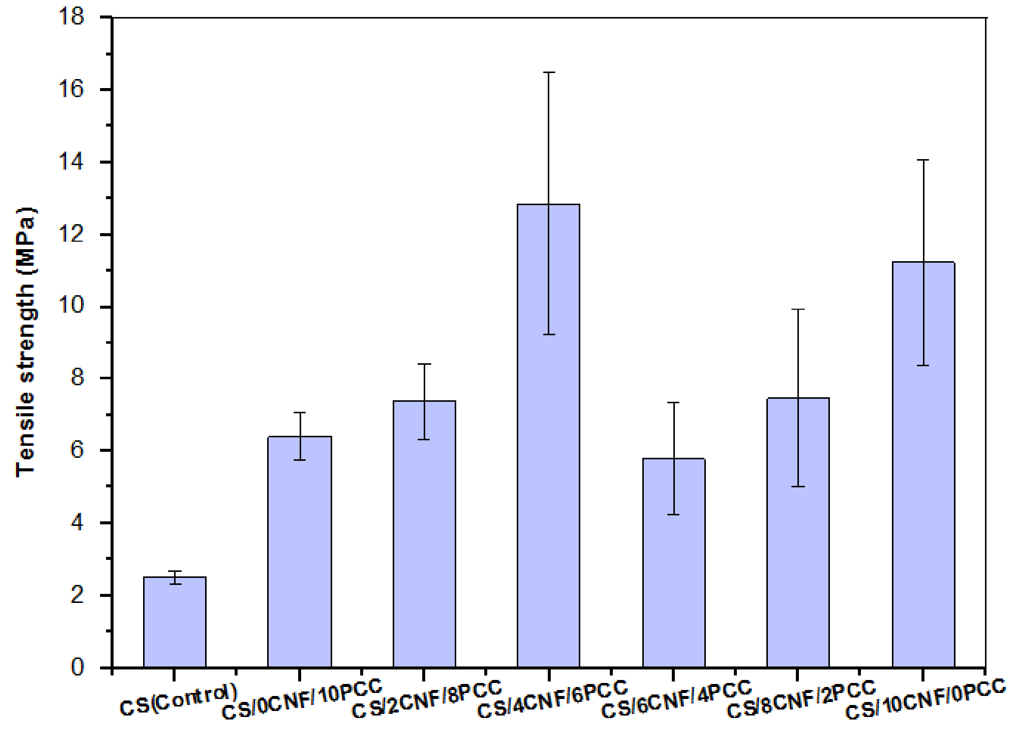
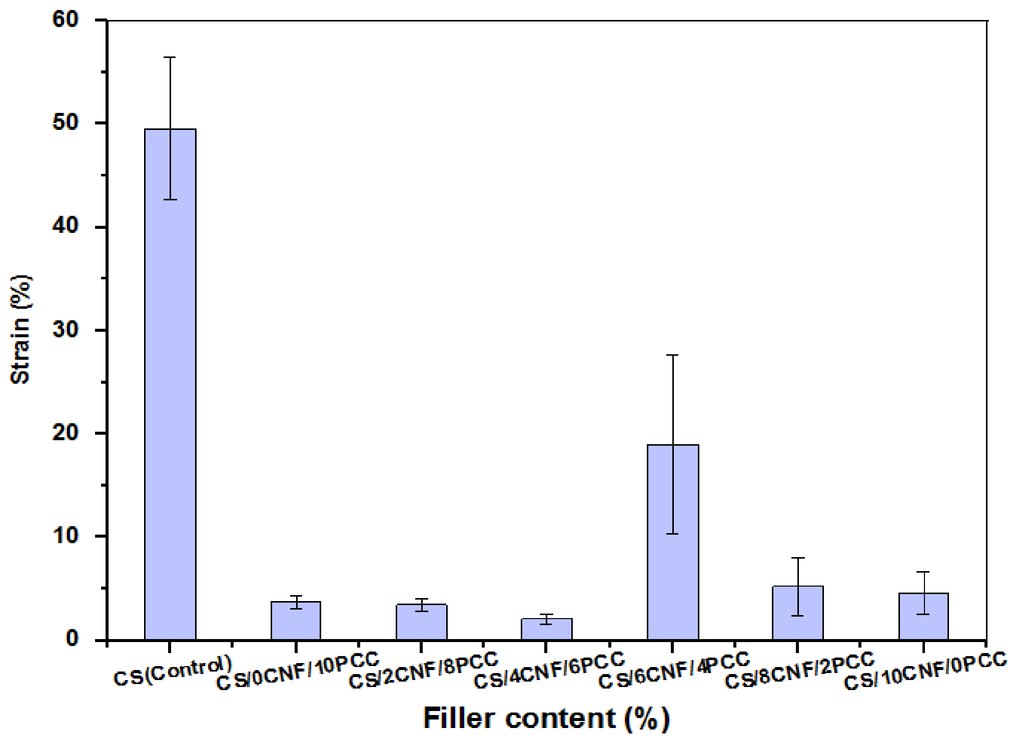


Figure 6. The tensile strength of CNF-ramie/PCC hybrid nanocomposites



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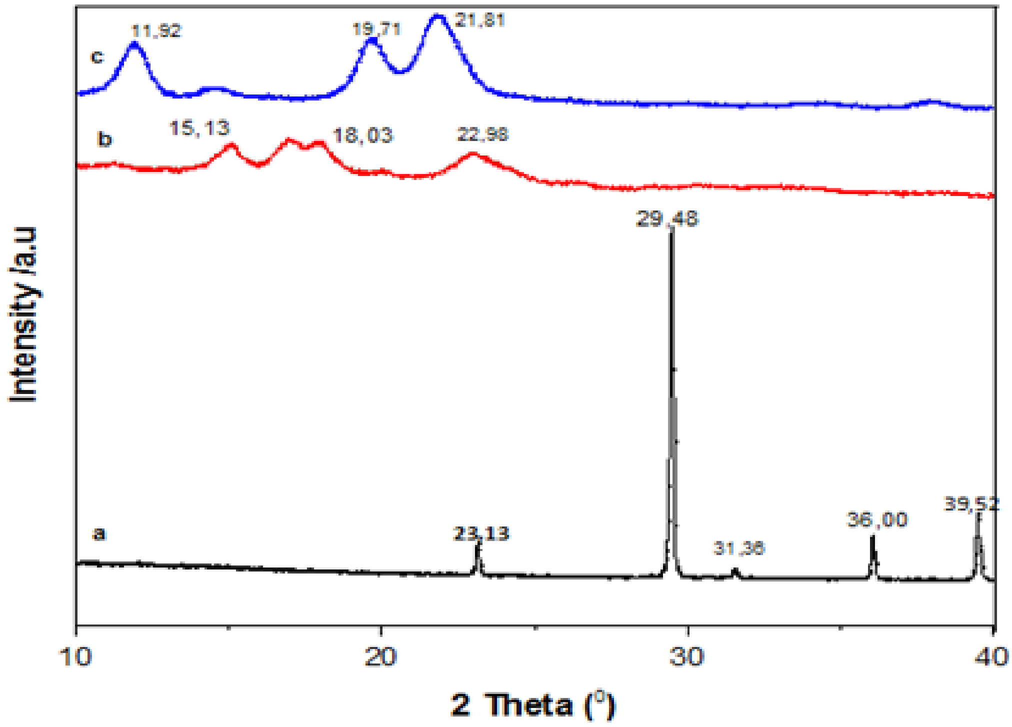


Figure 8. XRD curve pure materials of hybridnanocomposites, a) PCC, b) Starch, c) CNF- ramie

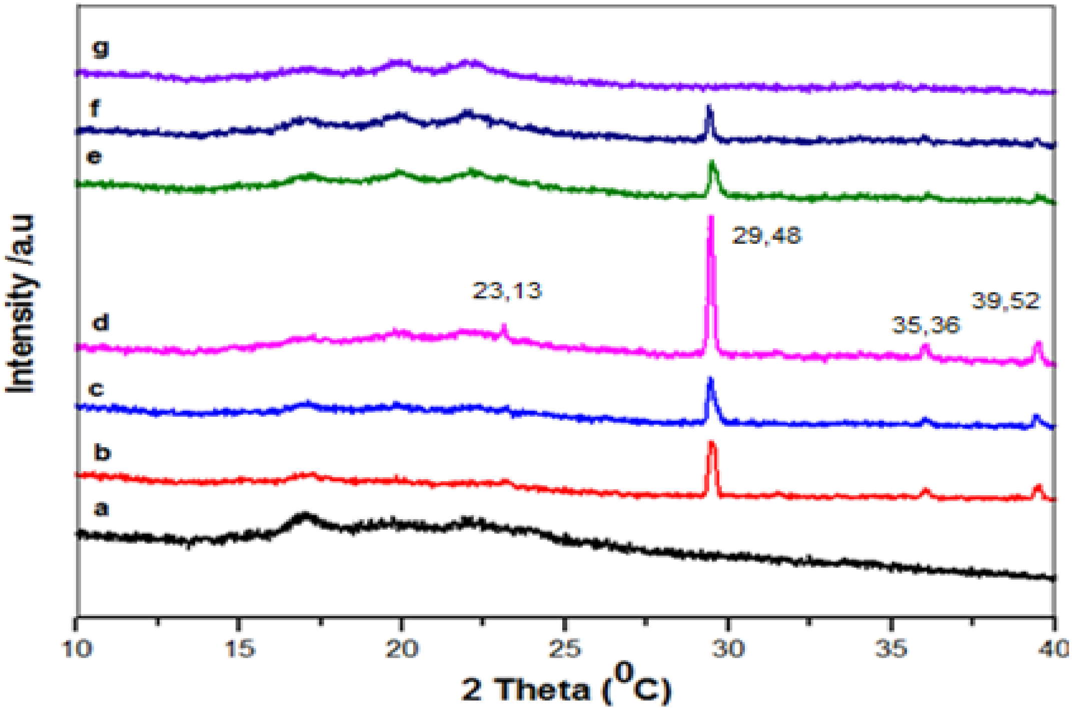


Figure 9. XRD curve of CNF-rami/PCC nano hybrid composites, a) CS (Control), b) CS/0CNF/10PCC, c) CS/2CNF/8PCC, d) CS/4CNF/6PCC, e) CS/6CNF/4PCC, f) CS/8CNF/2PCC and g) CS/10CNF/0PCC

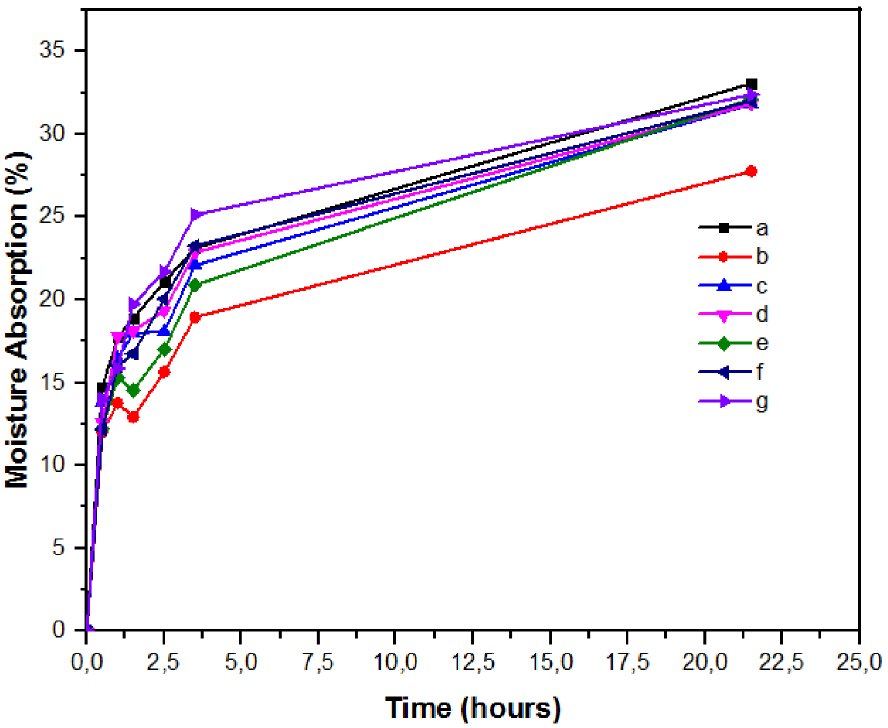
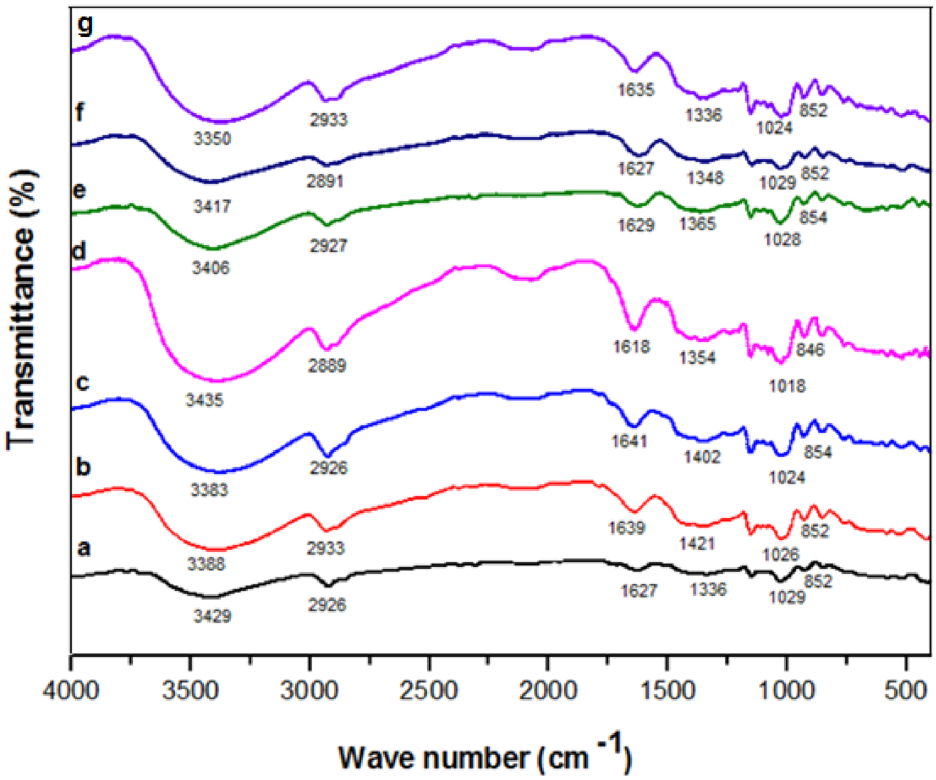


Figure 10. Moisture absorption of CNF-rami/PCC hybridnanocomposites, a) CS (Control), b) CS/0CNF/10PCC, c) CS/2CNF/8PCC, d) CS/4CNF/6PCC, e) CS/6CNF/4PCC, f) CS/8CNF/2PCC and g) CS/10CNF/0PCC



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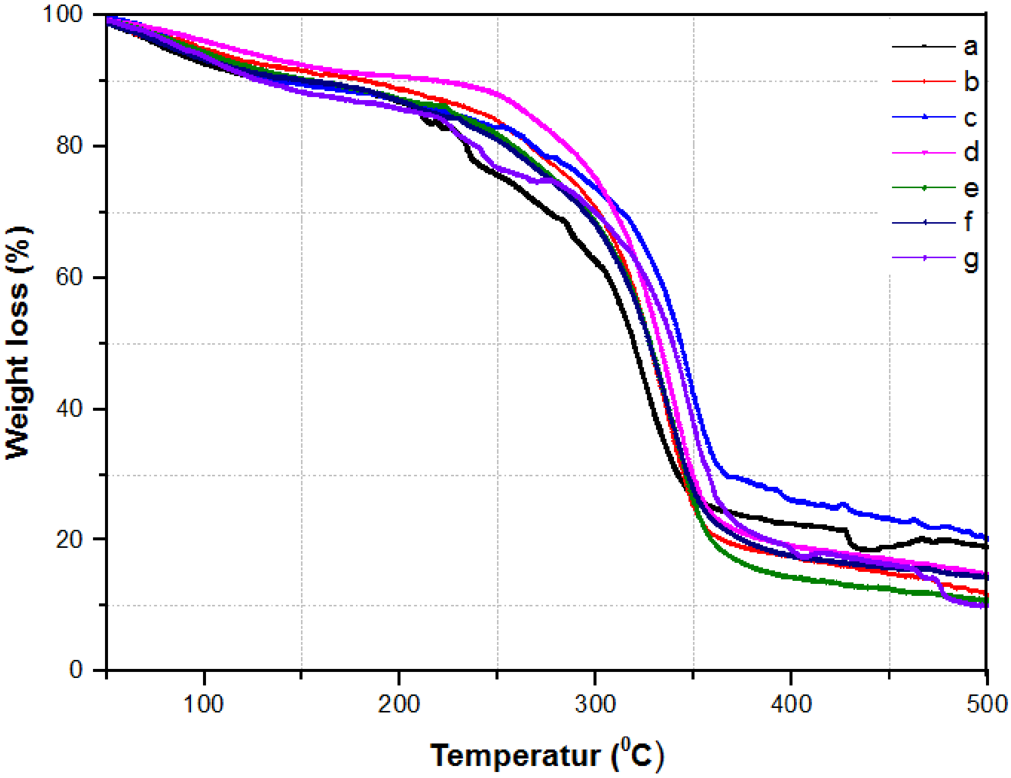

Figure 12. Thermogravimetric Analysis curve of CNF-ramie and PCC hybrid nanocomposites, a) CS (Control), b) CS/0CNF/10PCC, c) CS/2CNF/8PCC, d) CS/4CNF/6PCC, e) CS/6CNF/4PCC, f) CS/8CNF/2PCC and g) CS/10CNF/0PCC

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Table 1. Compositions of cassava starch (CS) and cellulose nanofibers (CNF)-ramie/Precipitated Calcium Carbonate (PCC) hybrid composites

Nanocomposites	Glyserol (g)	Starch (g)	CNF-ramie (g)	PCC (g)	Distilled water (ml)
CS (Control)	0,6	2	0	0	40
CS/0CNF/10PCC	0,6	2	0	1	40
CS/2CNF/8PCC	0,6	2	0,2	0,8	40
CS/4CNF/6PCC	0,6	2	0,4	0,6	40
CS/6CNF/4PCC	0,6	2	0,6	0,4	40
CS/8CNF/2PCC	0,6	2	0,8	0,2	40
CS/10CNF/0PCC	0,6	2	1	0	40

Table2. *Crystalline index* of CNF-rame/PCC nanocomposites hybrid

Variasi *filler*	***Crystaline index CI (%)***
CS (Control)	18.17
CS/0CNF/10PCC	12.36
CS/2CNF/8PCC	14.08
CS/4CNF/6PCC	30.76
CS/6CNF/4PCC	27.43
CS/8CNF/2PCC	26.42
CS/10CNF/0PCC	23.60

Table 3. Degradation temperatur of CNF-ramie/PCC hybridnanocomposites

CS Composites	Degradation Temperatur (^{0}C)			Massa Residue (%) at 500 ^{0}C
	$T_{10\%}$	$T_{30\%}$	$T_{50\%}$	
CS(Control)	144,07	276,36	319,18	19
CS/0CNF/10PCC	182,62	301,36	327,32	11,6
CS/2CNF/8 PCC	138,73	313,7	343,31	20,1
CS/4CNF/6PCC	221,35	309,47	332,45	14,9
CS/6CNF/4PCC	154,23	295,77	327,67	10,8
CS/8CNF/2PCC	151,62	294,4	327,32	14,3
CS/10CNF/0PCC	129,52	299,62	339	10