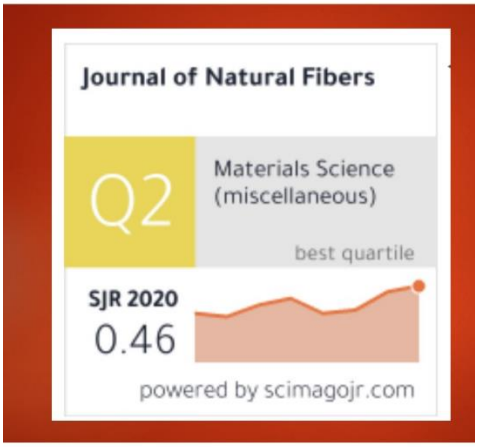
Bukti Korespondensi

Studies on Ramie cellulose microfibrils reinforced cassava starch composite: influence of microfibrils loading

Pengusul
Dr. Edi Syafri, ST, M.Si



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- **Q1** Impact Factor Best Quartile
- **3.760 (2021)** 5 year IF
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17-Mar-2018

Dear Mr Syafri:

Your manuscript entitled "Studies on Ramie cellulose microfibrils reinforced Cassava starch Composite: Influence of Microfibrils Loading", which you submitted to Journal of Natural Fibers, has been reviewed. The reviewer comments are included at the bottom of this letter.

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Once again, thank you for submitting your manuscript to Journal of Natural Fibers and I look forward to receiving your revision.

Sincerely,

Professor Kozlowski
Editor in Chief, Journal of Natural Fibers
ryszard.kozlowski@escorena.net

Reviewer(s)' Comments to Author:

Reviewer: 1

Comments to the Author
Abstract
- Review the phrase:
The addition of RCMF enhanced the crystallinity index (CI) up to 36.67% from 32%

Results
- Fourier Transformed Infrared Spectroscopy (FT-IR)
The correct word to denote the results ar "bands" not peaks. The word "peaks" you use in NMR for example.

- Cite the bands characteristics of cellulose.

-X-Ray
Explain better the influence of cellulose addition in the crystallinity

- The Table 5 isn´t in the list of Tables.

- Improve the discution, linking the results.

Questions
1. How is your explication for the below result.
TPS + 10% CMF - showed highest crystallinity but low FDT (Final degradation temperature). If the crystallinity is higher the thermayl estability would not be too?

Editor's Comments to Author:

Dear Editor

The Review WJNF-2018-0008, titled "**Studies on Ramie cellulose microfibrils reinforced Cassava starch Composite: Influence of Microfibrils Loading,** can be published in Journal of Natural Fibers, only after major revision.

General comments:

The text is well written, but the results could be deeply discussed.

Requests for improve the manuscript

Abstract

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We have incorporated all the comments and suggestions given by the you in the revised manuscript. Thank you for your valuable comments and corrections. I request you to please kindly accept our paper for publication in this Journal.

From: **Journal of Natural Fibers** <onbehalfof@manuscriptcentral.com>
Date: Sun, Mar 25, 2018 at 10:32 AM
Subject: Journal of Natural Fibers - Manuscript ID WJNF-2018-0008.R1 has been submitted online
To: edisyafri11@gmail.com

25-Mar-2018

Dear Mr Syafri:

Your manuscript entitled "Studies on Ramie cellulose microfibrils reinforced Cassava starch Composite: Influence of Microfibrils Loading" has been successfully submitted online and is presently being given full consideration for publication in Journal of Natural Fibers.

Your manuscript ID is WJNF-2018-0008.R1.

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Sincerely,
Journal of Natural Fibers Editorial Office

Manuscripts I Have Co-Authored

STATUS	ID	TITLE	CREATED	SUBMITTED
ADM: Maciejowska, Izabela • Accept (24-Apr-2018)	WJNF-2018-0008.R1	Studies on Ramie cellulose microfibrils reinforced Cassava starch Composite: Influence of Microfibrils Loading *Files Archived*	25-Mar-2018	25-Mar-2018

Publish

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Studies on Ramie cellulose microfibrils reinforced cassava starch composite: influence of microfibrils loading

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[b]Department of Agricultural Product Technology, Andalas University Kampus Limau Manis, West Sumatra, Indonesia;
[c]Department of Mechanical Engineering, Andalas University Kampus Limau Manis, West Sumatra, Indonesia;
[d]Department of Mechanical Engineering, Kamaraj College of Engineering and Technology, Virudhunagar, India;
[e]Department of Mechanical Engineering, Ramaiah Institute of Technology, Bengaluru, India

ABSTRACT

Composites were fabricated from Ramie cellulose microfibrils (RCMF) with cassava starch as matrix and glycerol as a plasticizer. Different composites were fabricated with microfibrils loadings of 0, 2, 4, 8, and 10 wt%. The Particle Size Analyzer results show the average size of RCMF as 1.573 µm. The addition of RCMF considerably influenced the physical, crystalline, thermal, and tensile properties of composites. The addition of RCMF enhanced the crystallinity index (CI) from 32% to 36.67%. Thermogravimetric analysis and tensile test results showed improvement in thermal stability and tensile strength of composite up to 6 wt% microfibrils addition.

摘要

以Ramie纤维素微纤维（RCMF）为原料，以木薯淀粉为基质，甘油为增塑剂，制备了复合材料。用微纤维负载量分别为0、2、4，8和10WT%制备不同的复合材料。粒度分析结果表明，Ramie纤维素微纤维的平均粒径为1.573µm。苎麻纤维素微纤维的加入显著影响了复合材料的物理、结晶、热、拉伸性能。苎麻纤维素微纤维的加入使结晶度指数（CI）从32%提高到36.67%。热重分析和拉伸试验结果表明，复合材料的热稳定性和拉伸

KEYWORDS

Cassava starch; Ramie cellulose microfibrils; thermal stability; tensile strength

关键词

木薯淀粉; 纤维素微纤维; 热稳定性; 抗拉强度

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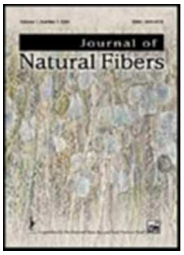

Studies on Ramie cellulose microfibrils reinforced Cassava starch Composite: Influence of Microfibrils Loading

Journal:	*Journal of Natural Fibers*
Manuscript ID	WJNF-2018-0008.R1
Manuscript Type:	Short Communication
Keywords:	Cassava starch, Ramie cellulose microfibrils, Thermal stability, Tensile strength, XRD, FTIR

Reviewer(s)' Comments to Author:

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Abstract

Composites were fabricated from Ramie cellulose microfibrils (RCMF) with cassava starch as matrix and glycerol as a plasticizer. Different composites were fabricated with microfibrils loadings of 0,2, 4, 8 and 10wt%. The Particle Size Analyzer results show the average size of RCMF 1.573 µm. The addition of RCMF considerably influenced the physical, crystalline, thermal, and tensile properties of composites. The addition of RCMF enhanced the crystallinity index (*CI*) from 32% to 36.67%. Thermogravimetric analysis and tensile test results showed that improvement in thermal stability and tensile strength of composite upto 6 wt % microfibrils addition.

Key words: Cassava starch, Ramie cellulose microfibrils, Thermal stability, Tensile strength.

INTRODUCTION

The inventions of biodegradable materials are necessary for overcoming today environmental pollution so, the many of the researchers focusing on natural fiber reinforced polymer composites instead of synthetic fiber reinforced composites (Senthamaraikannanet al. 2015). Even move towards the natural fibers still most of the researchers using matrix material as synthetic resins so, the resultant composites are partially biodegradable material. To make the completely bio degradable material, we have to move towards the biodegradable matrix. Cassava (*Manihotesculenta*) starch is one of the commercially available biodegradable matrices throughout the world (WirongrongTongdeesoontorn et al. 2011). However, the cassava starch is brittle in natures which resulting poor mechanical properties, to overcome this difficulty, plasticizers are added to the starch which may decrease the brittleness of the

material and enhance the process ability, on the other hand which may reduce the degradation temperature of starch (Chang et al. 2006). Addition to these, starch has higher water absorption nature. Reinforcing of natural fiber is one of the efficient methods to improve the performance of the starch based composite. The hydrophilic character of natural fiber may disturb the bonding between fiber and matrix which may be considerable reduced by surface modification of fibers (Saravanakumaret al. 2014). The fiber size also plays the important role in the composite properties (Sanjayet al. 2018; Kathiresan & Sivaraj 2015). In this study, characterised the Ramie cellulose microfibrils reinforced cassava starch composites through TGA, FTIR, XRD, SEM, Tensile testing and Water absorption test for checking the effect of microfibrils loading on the physical, chemical, thermal and tensile properties.

MATERIALS AND METHODS

Materials

Cassava starch (Cap Tani, Indonesia), Glycerol (PT Cisadane Raya Chemicals, Tangerang Indonesia) and Ramie cellulose microfibrils were used to manufacture the composite. NaOH, KOH, $NaClO_2$ were utilized for the preparation of Ramie cellulose microfibrils.

Preparation of Ramie cellulose microfibrils

Various processes involved in preparation of Ramie cellulose microfibrils (CMF) is illustrated in Fig.1 and Fig.2 presents different forms of Ramie.

Composite Preparation

The procedure followed by the Tongdeesoontorn et.al was adopted with some modification for preparation of Thermoplastic starch (TPS) composites (Fig.3) (WirongrongTongdeesoontorn et al. 2011). Cassava starch of 10 grams and glycerol of 2.5 grams (plasticizer) were dissolved in the 140 ml of water then 0, 0.2, 0.4,0.6,0.8,1 grams of Ramie CMFs were mixed with this solution for making different composite varieties noted in

Table 1. The (Cassava starch / glycerol / CMF) solutions were heated upto 100 ^{0}C with continuous stirring (350 rpm) to gelatinization then poured into a glass mould (20 x 20 x 0.5 cm^3). The mould set up is placed in ultrasonic bath for removing voids from composite then positioned in an oven at 40^{0}C for 5 hours.

Particle Size Analyzer

The particle sizes of Ramie cellulose microfibrils were finalized by Particle Size Analyzer (DelsaNano C) with the capacity to measure 0.6 nm - 30 mm. The samples were mixed with deminaralized water then laser light passed through this solution and the scattering angle was measured to find the particle size. Experiments were done in the 25°C and Delsa Nano software was used for data processing.

Thermogravimetric analysis (TGA)

The thermal stability of composites were measured by the TGA 4000 (Perkin Elmer) instrument. The samples were heated from room temperature to 400^{0}C with the heating rate of 10 °C/ min. Nitrogen gas was passed with the mass flow rate of 40 ml/min during the experimentation (Prithiviraj et al. 2016).

Fourier Transformed Infrared Spectroscopy (FTIR)

The FTIR (Perkin Elmer) spectrums of composites were documented to find the structural difference between composites. The spectrums were recorded from 4000 to 600 cm^{-1} Wave number range at room temperature with the resolution of 4cm^{-1}(Kathiresan et al. 2016).

X-ray diffraction (XRD) analysis

Crystallinity index (*CI*) is the important parameter which influences the tensile properties of the composites. In order to investigate the effect of microfibrils loading on the crystallinity index of the composites, the X-ray diffractograms of composite were recorded by PANalytical's X-ray diffractometer in the 2θ range of 10.0181° to 99.9781°. The experimentation conditions were Generator Settings (30 mA, 40 kV), continuous scanning with the step size of 2θ=0.001° and Measurement Temperature (25°C).

The crystallinity index (CI) was assessed through the following equation (Manimaranet al. 2018)

$$I_c = \left(1 - \frac{I_{am}}{I_{002}}\right) \times 100\,\% \quad (1)$$

Where I_{002} is the intensity of crystalline peak, and $I_{am}$is the intensity of amorphous peak in the XRD spectrum

Tensile testing

The tensile strength, young's modulus and strain (%) of composite were measured by a Com-Ten of 95T Series tensile testing machine. The tests were conducted as per the guidelines of ASTM D 638-1(WirongrongTongdeesoontornet al.2011). All the trials were carried out at crosshead speed of 2 mm/min in the ambient temperature.

RESULTS AND DISCUSSION

Particle Size Analyzer

The combined Pulping, bleaching and grinding process converted Ramie cellulose fiber into the Ramie cellulose microfibrils with the average size of 1.573 μm which is used as reinforcement in composites. Fig.4 (a) shows the size distribution of microfibrils.

Thermogravimetric analysis (TGA)

The TGA curves of the composites were presented (Fig.4 (b)) to understand the effect of Ramie cellulose microfibrils on the thermal stability of composites. From Table.2, it was established that the initial degradation temperature (IDT), Final degradation temperature (FDT) and Inflection point of composites were increased upto 6 wt.%microfibrils addition after that, reduced. The first phase was happened between 90 -115^{o}C, which is related to the loss of moisture and related impurities in the composites (Vajja Sadanand et al.2017). The second phase of mass loss occurred between 280^{o}C to 345^{o}C which may be due to the decomposition of cellulose, Hemicellulose and lignin in the composite (Alireza Ashori & Reza Bahrami 2014) The final, tailing phase from 345° to 500°C indicated the degradation of the charred residue.

Fourier Transformed Infrared Spectroscopy (FT-IR)

The chemical functional groups existing in the Ramie cellulose microfibrils reinforced cassava starch composites were investigated by FT-IR (Fig. 5 (a)). FT-IR spectra of all the specimens have the similar bands which show that cassava starch is not chemically affected or modified by the glycerol or the fiber loading during the composite manufacturing (Edi Syafri et al.2017). From Fig. 6, it can be detected that five distinct bands were existing in all the samples nearby 3296, 2926, 1647, 1353 and 1011 cm^{-1}(Senthamaraikannan et al. 2015).. The band around 3296 cm^{-1} specify the occurrence of O–H groups in cassava starch and microfibrils which illustrate that composites are reactive to water molecules because of the existence of hydroxyl groups (Kishanji et al. 2013). The band raised nearby 2926 cm^{-1} belongs to the C–H stretching vibrations. The band around 1647 cm^{-1} validates the occurrence of water in the composites The bands formed between 1353 cm^{-1} and 1647 cm^{-1} is an indication of the occurrence of strong and broad -C-O stretching, which is the alcohol

groups of cellulose (Edi Syafri et al. 2017). The bands around 1353 cm^{-1} and 1010 cm^{-1} are corresponding to the C-OH bending vibrations. Table 3 presents the band positions and assignments of chemical groups in the Ramie cellulose microfibrils reinforced cassava starch composites.

X-ray diffraction (XRD) analysis

X-ray diffraction (XRD) analyses were executed to find out the influence of microfibrils content on the crystallinity index of the composites. The XRD spectrums of the Ramie cellulose microfibrils reinforced cassava starch composites are shown in Figure 5 (b). Two important peaks were detected at $2\theta = 16.6^{o}$ (amorphous phase) and $2\theta = 22.3^{o}$ (crystalline phase) indicating the presence of cellulose type-I which signifies its semi crystalline nature. The minor peak at $2\theta = 16.6°$ is attributed to (2 0 0) crystallographic plane contributing towards amorphous fraction and the major peak at $2\theta = 22.3°$ is ascribed to (0 0 2) crystallographic plane showing the occurrence of both amorphous and crystalline fractions. From Table 4, it was recognized that the composite with a higher microfibrils content have a higher crystallinity index. The Ramie cellulose microfibrils are orientated materials than cassava starch so, which influenced the crystallinity of the composites (Edi Syafri et al.2017).

Tensile testing

The tensile behaviour of Ramie cellulose microfibrils reinforced cassava starch composites are shown in Table 5. It is detected that the tensile strength and young's modulus of composites enhanced with addition of microfibrils content up to 6 wt.%, after that, decreased which may be due to the lack of interfacial bonding between the microfibrils and cassava starch above 6 wt.% microfibrils addition(WirongrongTongdeesoontorn et al. 2011). If the microfibrils content more than 6 wt.%, the cassava starch matrix was not sufficient to dissolve the microfibrils so, that tensile strength and young's modulus of composites were

reduced. However, strain rate of composites decreased with addition of microfibrils content. The 6 wt.% microfibrils added composite delivered favourable properties than other composites which directing enhanced stress transfer between the microfibrils to the matrix and interfacial bonding in this composite (Sanjay et al. 2018)

Conclusion

Ramie cellulose microfibrils reinforced composites were produced with the main aim of create completely biodegradable composites with better performance. The addition of CMFs considerably improved the performances such as thermal stability, tensile strength and crystallinity index of the composites. However, no noteworthy chemical functional group modification found on the FTIR spectrums.The tensile strength of the composites were improved from 1.65±0.21MPa to 7.41±1.63 (6 wt%) and tensile stability enhanced from 281.46 to 291.41◦C (6 wt%).

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Table Captions:

Table 1: Composition of Ramie cellulose microfibrils reinforced cassava starch composites

Table 2: Thermal degradation parameters of RCMF reinforced cassava starch composites

Table 3: Peak positions and assignments of chemical groups in the Ramie cellulose microfibrils reinforced cassava starch composites

Table 4: The crystallinity index of RCMF reinforced cassava starch composites.

Table 5: Mechanical properties of the Ramie cellulose microfibrils reinforced cassava starch composites

Table 1: Composition of Ramie cellulose microfibrils reinforced cassava starch composites

Composite	Composition (g/140 ml distilled water)		
	Cassava starch (g)	Glycerol (g)	Ramie CMF (g)
TPS+0%CMF	10	2.5	0
TPS+2%CMF	10	2.5	0.2
TPS+4%CMF	10	2.5	0.4
TPS+6%CMF	10	2.5	0.6
TPS+8%CMF	10	2.5	0.8
TPS+10%CMF	10	2.5	1

Table 2: Thermal degradation parameters of RCMF reinforced cassava starch composites.

Composite	IDT (◦C)	FDT (◦C)	Inflection point (◦C)	% Char content	% Moisture content
TPS+0%CMF	281.46	336.61	310.62	11.56	9.42
TPS+2%CMF	285.29	338.71	316.67	12.15	10.71
TPS+4%CMF	286.87	340.14	316.92	11.99	11.27
TPS+6%CMF	291.41	342.42	318.98	12.63	9.72
TPS+8%CMF	288.78	338.77	316.42	12.56	10.31
TPS+10%CMF	271.71	339.78	313.82	12.67	10.73

Table 3: Peak positions and assignments of chemical groups in the Ramie cellulose microfibrils reinforced cassava starch composites

Peak positions (Wavenumber (cm^{-1}))							**Allocations**
Ideal FTIR peak position	**TPS+0% CMF (a)**	**TPS+2% CMF (b)**	**TPS+4%CMF (c)**	**TPS+ 6% CMF (d)**	**TPS+8% CMF (e)**	**TPS+10% CMF (f)**	
3600–3100	3296.31	3263.64	3292.84	3293.55	3290.61	3294.04	O-H stretching vibration of cellulose molecules
2960–2850	2926.35	2926.29	2926.49	2926.19	2926.34	2925.37	C-H stretching vibrations
1740–1600	1647.18	1647.41	1647.33	1647.49	1647.05	1646.47	Presence of water
1320	1353.12	1353.88	1353.23	1353.85	1353.38	1353.01	C-OH bending vibrations
1050–1020	1011.43	1010.27	1012.27	1011.39	1011.16	1010.99	C-OH stretching

Table 4: The crystallinity index of RCMF reinforced cassava starch composites.

Composite	Crystallinity Index (%)
TPS+0%CMF	23.91
TPS+2%CMF	26.24
TPS+4%CMF	28.46
TPS+6%CMF	34.56
TPS+8%CMF	35.72
TPS+10%CMF	36.67

Table 5: Mechanical properties of the Ramie cellulose microfibrils reinforced cassava starch composites

CMF Ramie Contents	Tensile Strenth (MPa)	Tensile Modulus (MPa)
TPS+0%CMF	1,65	174,61
TPS+2%CMF	5,65	188,45
TPS+4%CMF	6,37	235,31
TPS+6%CMF	7,41	261,22
TPS+8%CMF	5,25	234,82
TPS+10%CMF	4,88	181,81

Figure captions:

Figure 1. Preparation of Ramie cellulose microfibrils

Figure 2. (a) Chopped Ramie fibers, (b) Pulped Ramie powder, (c) Bleached Ramie powder

Figure 3. (a) Cassava starch / glycerol / CMF solution (before gelatinization), **(b)**Poured into the mould, **(c)** Mould Placed in the ultrasonic bath **(d)** Composite (After removed from mould), and **(e)** SEM images of Ramie cellulose microfibrils reinforced cassava starch composites

Figure. 4 (a). Distribution of size CMF Ramiemicrofibrils

Figure4 (b). TGA of RCMF reinforced cassava starch composites

Figure 5 (a). FTIR analysis of Ramie cellulose microfibrils reinforced cassava starch

Figure 5 (b). XRD analysis of Ramie cellulose microfibrils reinforced cassava starch

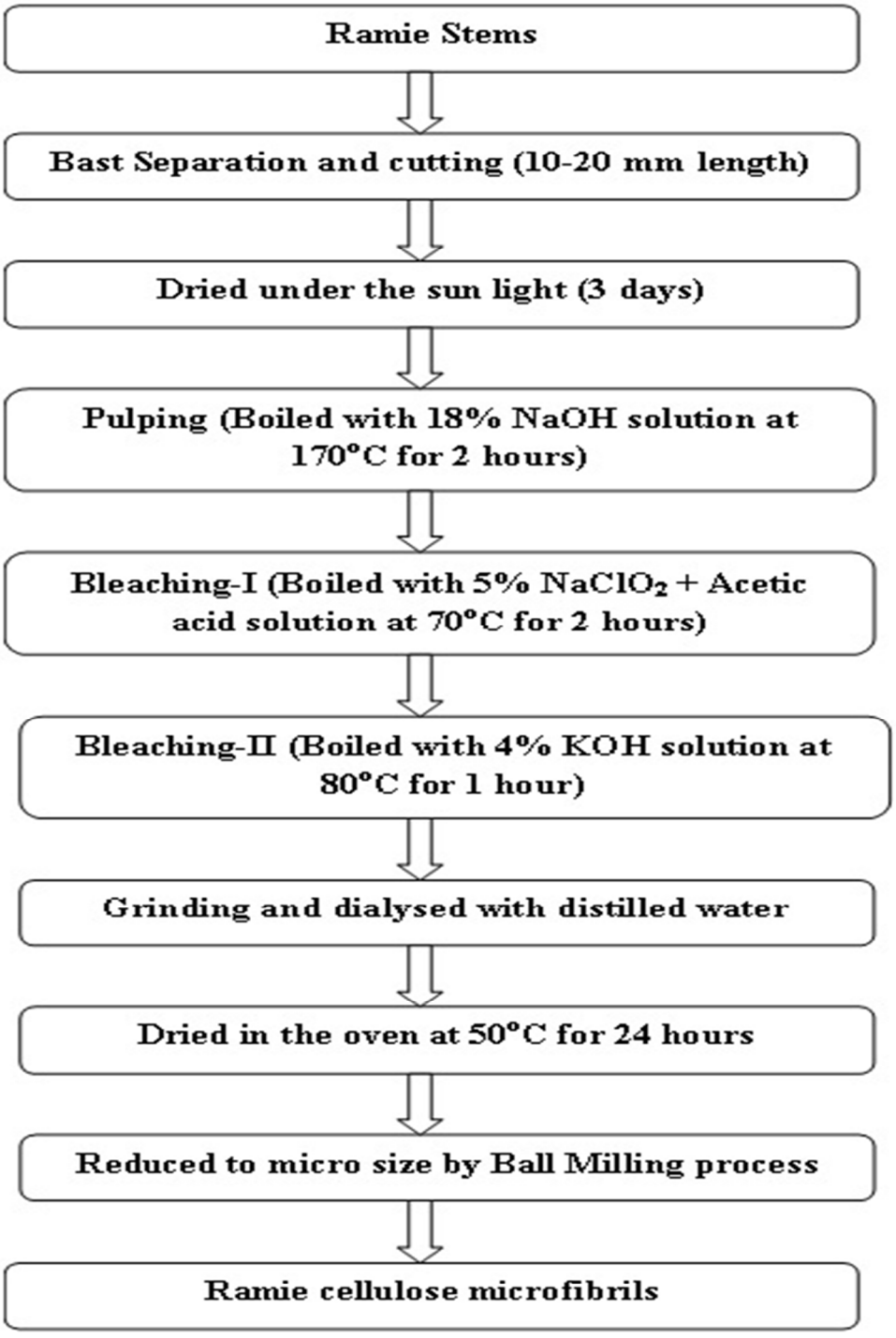
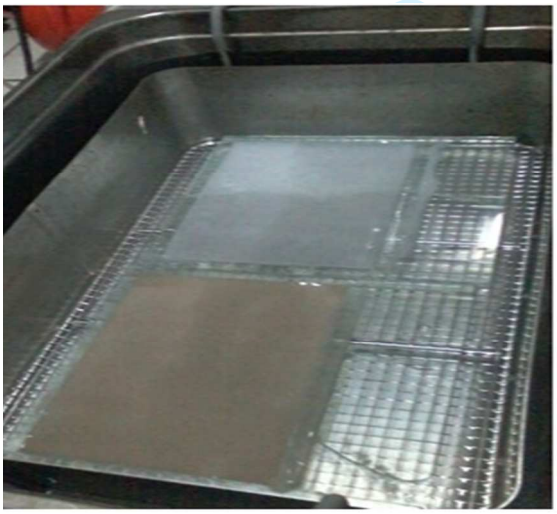


Figure 1. Preparation of Ramie cellulose microfibrils

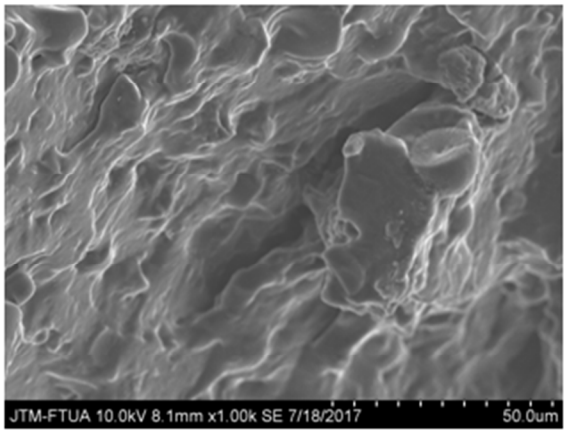
Figure 2. (a) Chopped Ramie fibers, (b) Pulped Ramie powder, (c) Bleached Ramie powder

(a) **(b)**

(c) **(d)**



(e)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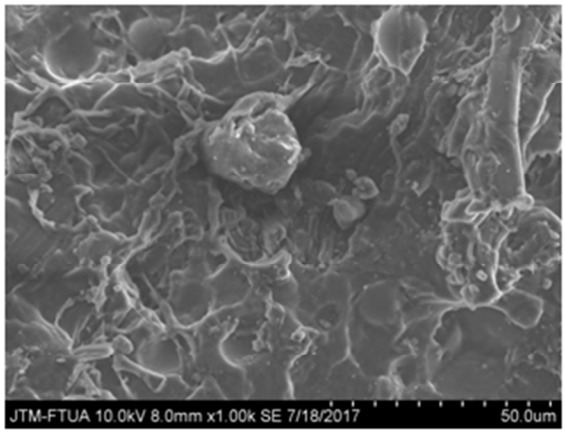
50.0um


JTM-FTUA 10.0kV 8.0mm x1.00k SE 7/18/2017
50.0um

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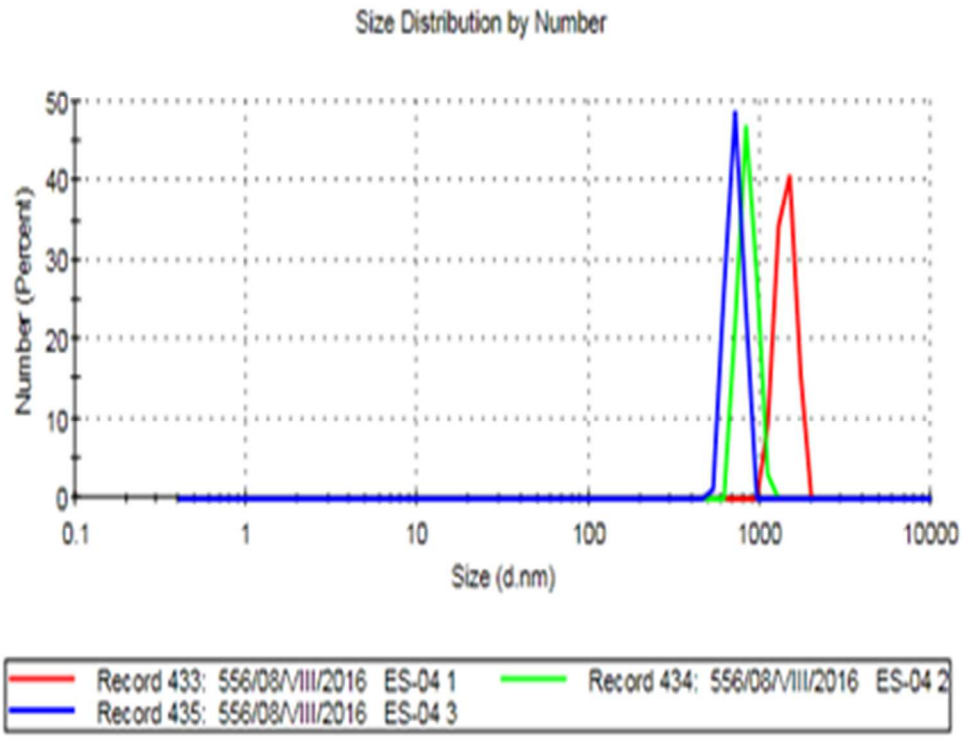

Figure 4 (a). Distribution of size CMF Ramie microfibrils

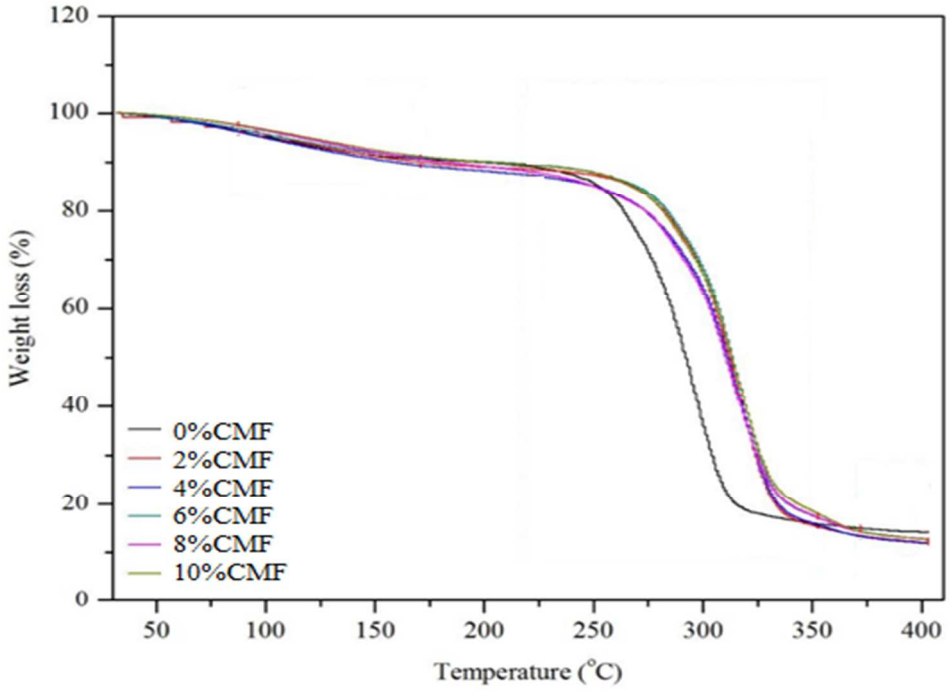

Figure 4 (b). TGA of RCMF reinforced cassava starch composites

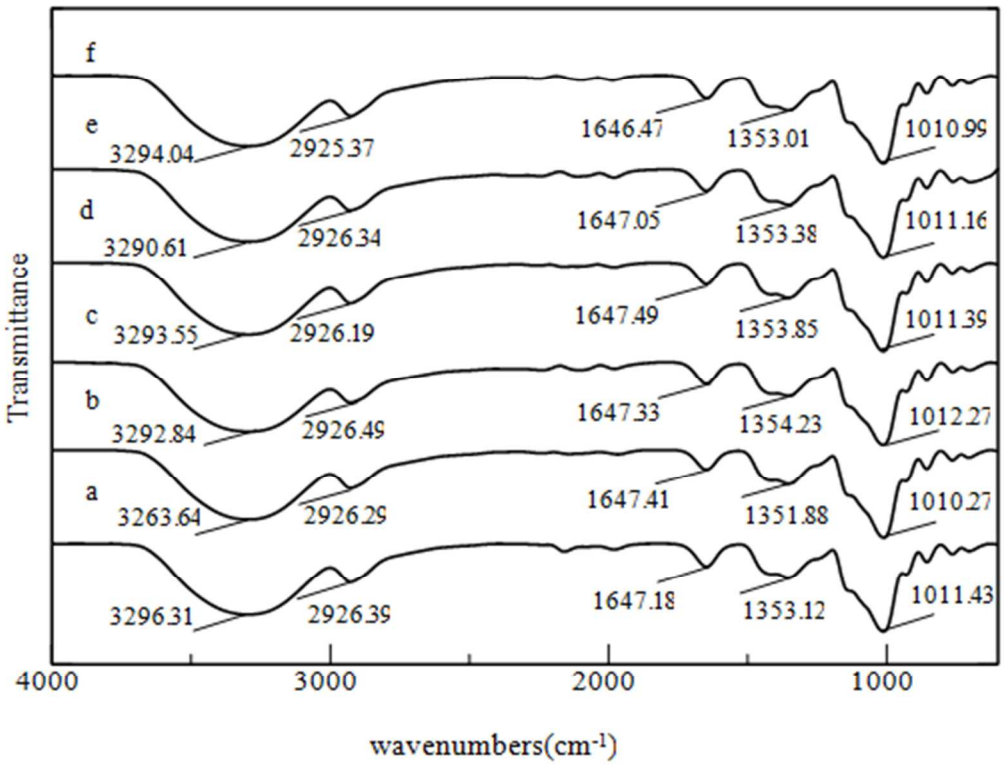

Figure 5 (a). FTIR analysis of Ramie cellulose microfibrils reinforced cassava starch

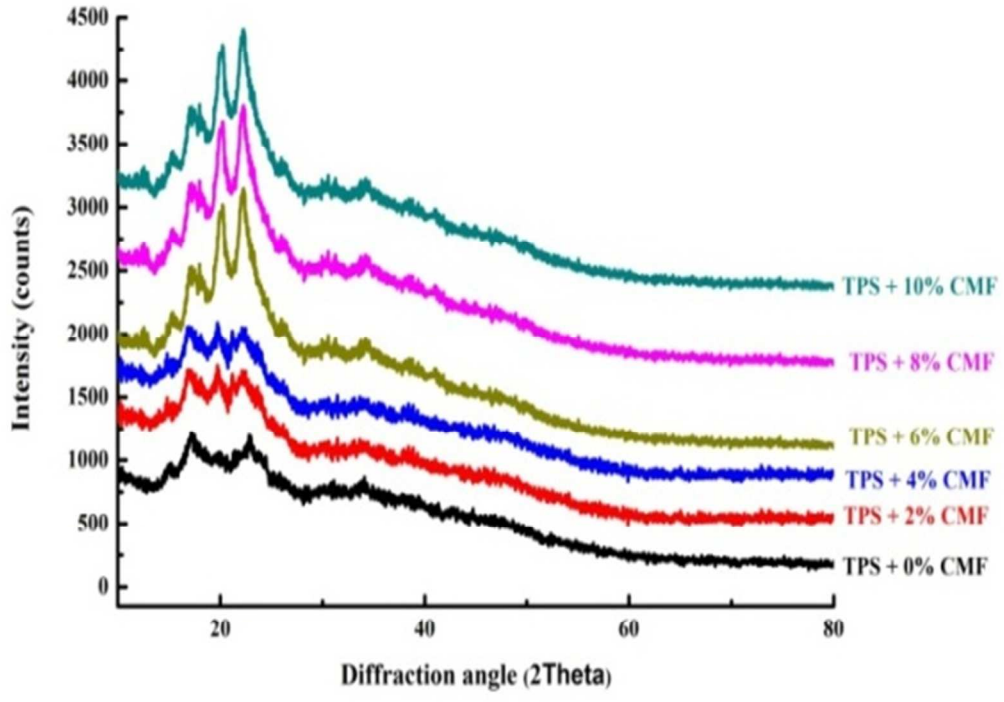

Figure 5 (b). XRD analysis of Ramie cellulose microfibrils reinforced cassava starch