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THERMAL ANALYSIS AND MICROSTRUCTURE OF CARBON BLACK NANOPARTICLES FROM COCONUT SHELLS

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ABSTRACT

Carbon Black (CB) is widely used as a filler in rubber materials. CB has been obtained from the thermal cracking process of petroleum hydrocarbons. With the depletion of petroleum reserves, it is necessary to substitute carbon black using natural materials such as coconut shells. This research studied the thermal and microstructure properties of Coconut Shell Nanoparticles (CSNP). CSNP was synthesized by the ball mill method and coprecipitation method. Synthesis of Coconut Shell (CS) by coprecipitation method with various solvents affects the thermal properties and microstructure of the resulting carbon black. Morphological results showed that most of the carbon pores were formed, and the dominant elements in the sample were carbon and oxygen. The appearance of the absorption band, which is characteristic of CB in CS samples, was confirmed from the FTIR results. In addition, from XRD analysis, the particle size of $CSNP_1$, $CSNP_2$, $CSNP_3$ samples was (33.2, 52.92, and 54.98) nm with an amorphous structure. The endothermic peak is associated with phase reduction and decomposition reactions. The thermal stability of nanoparticle coconut shells is better than coconut shells.

Keywords: coconut shell, carbon black, nanoparticle, coprecipitation.

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1. INTRODUCTION

Carbon black (CB) is an important material to improve rubber-based materials' physical and mechanical properties. Thus, various industries need it [1]. CB is obtained from the thermal cracking process of petroleum hydrocarbons. With the depletion of petroleum reserves, it is necessary to substitute carbon black using non-oil and gas natural materials. The natural materials used have criteria for low prices, abundant availability, and being environmentally friendly [2].

Globally, various studies have concentrated on agricultural waste such as coconut shells, oil palm boiler ash, and oil palm empty fruit bunch ash. These wastes occur as low-cost carbon reinforcements and fillers in the development of metal and polymer composites which are structural composites for aircraft and automobile applications [3]–[9]. Nowadays, scientists and engineers conduct research based researching natural resources to supply energy demand in industrial processes and advanced technology [10]. Natural fillers such as excellent specific strength, high toughness, low cost, and increased energy recovery [11].

Coconut Shell (CS) is a promising material to replace carbon black as a nanofiller. It is because of CS which has a high content of carbon black[12]. Pyrolysis is a process to convert CS into carbon. In the pyrolysis process, the elements such as hydrogen (H) and oxygen (O) will be lost, and carbon (C) will remain. The carbon content in CS is 57.11%, oxygen is 42.67%, and other materials are 0.23%. The composition of CS such as cellulose, lignin, hemicellulose containing C, O, H, and N atoms [13].

Coconut shell as a substitute for CB in rubber-based materials shows a higher tensile modulus [14]. Likewise, CS is filler in bioplastic polymers, increasing mechanical strength and reducing mass [15]. The particle size of the filler affects the improvement of the composite properties. The nanoparticles are strong reinforcement in polymers to improve thermal and mechanical properties [16]–[18]. The thermal efficiency of nanomaterials is better than bulk materials. The thermal efficiency of bulk materials can be increased by enclosing nanoparticles into it [19]. Several methods of synthesis of Coconut Shell Nanoparticle (CSNP) include the Wet-Stirred Media Milling method, mechanical milling, coprecipitation [20]–[23].

Chemical deposition techniques, such as coprecipitation, are rarely used in CSNP synthesis. The coprecipitation method can control the structure, morphology, and composition of nanomaterials if each reaction step is appropriately controlled. Essential parameters to determine the nature of the final product of nanoparticles are reagent concentration, time and temperature, pH value of the solution, and surfactant used [24]. Therefore, this research was aimed to study the thermal and microstructure properties of CS nanoparticles synthesized by the ball mill method and coprecipitation by varying the molarity of the solvent used.

2. MATERIALS AND METHODS

2.1 Material

This research was carried out in the laboratory of Universitas Negeri Medan. The materials used are

Coconut shells, HCl (5M, 6M, 7M), NH_4OH Merck Pro Analis, PEG 6000 Merck, Aquades.

2.2 Methods

2.3 Synthesis of Coconut Shells Nanoparticles

The CS was crushed and put in the oven at 200°C for 1 hour. CS was ground with Planetary Ball Mill Retsch PM 200 for 2 hours with a speed rotation of 250 rpm. CSNP synthesis was carried out by the coprecipitation method. CS was mixed and stirred with each variation of 5M, 6M, 7M HCl solution at 70°C for 40 minutes and labeled in Table-1. Then filtered and mixed with 3M NH_4OH for 40 minutes at 70°C using a magnetic stirrer and then washed repeatedly using distilled water to establish a neutral pH. CSNP was dried at 70°C for 4 hours.

Table-1. Sample Label.

No	Sample Label	Material
1.	CS	Coconut shell
2.	$CSNP_1$	Coconut shell is treated with 5 M HCl
3.	$CSNP_2$	Coconut shell is treated with 6 M HCl
4.	$CSNP_3$	Coconut shell is treated with 7 M HCl

3. RESULT AND DISCCUSIONS

3.1 X-Ray Diffraction (XRD) Characterization

At a certain frequency from infrared radiation, resulting in bond vibrations within the molecule. Multiple signals are very easy to use to identify a particular type of bond in a 500 cm-1) usually contains very complex absorbent forms.

X-ray diffraction analysis (XRD) was performed with a Shimadzu 6000 type Goniometer. In powdered samples, the characterization was carried out using a Cu/Kα1 X-ray source with a wavelength = 1.54056 nm. To identify the diffraction pattern of the synthesized carbon material, the resulting diffractogram was stack up with the Joint Committee on Powder Diffraction Standard database, PDF Card 41-1487. Crystal size analysis (D) on carbon material is carried out using the equation:

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

Where D is the crystal size (Å), the X-ray wavelength used for CuKα radiation (λ = 1.54056 nm nm), θ is the Bragg angle, κ is the Scherrer constant that having a general value of 0.9, and β is the peak half-full width (FWHM) in radians. Figure-1 shows the XRD diffraction.

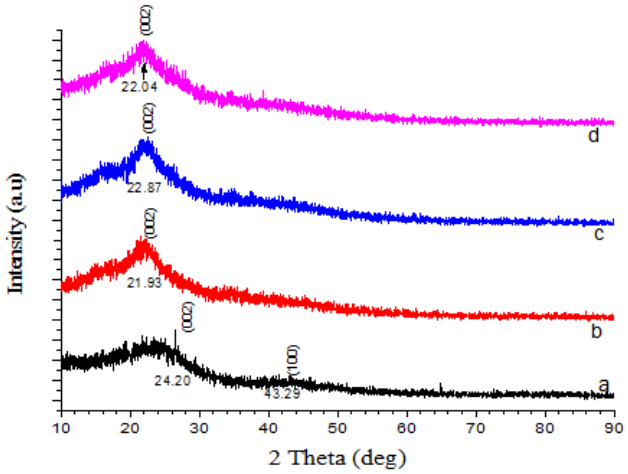

Figure-1. FTIR graph of: a). OPBA calcination and ball mill, b). OPBA coprecipitation, c). modification OPBA with added PEG 6000.

Figure-1 shows the value of dhkl in the form of wide-angle range and sloping peaks. This situation indicates that the sample of CB nanoparticles from the CS has an amorphous structure [25]–[27]. In general, samples derived from organic or natural materials usually have an amorphous solid structure. In addition to determining the state of the material structure, XRD characterization results are also used to determine the particle diameter using Scherer calculations and determine the material sample's angle. The particle size is presented in Table-2. The escalation in particle size with an increase in solvent molarity may be influenced by the concentration of the solvent, which causes agglomeration.

Table-2. The angle and size of the crystalline diameter were based on the XRD pattern.

No	Sample Code	2θ (002) (°)	Particle Size (nm)
1	CS	24,204	None
2	$CSNP_1$	21,931	33,2
3	$CSNP_2$	22,872	52,92
4	$CSNP_3$	22,045	54,98

3.2 Scanning Electron Microscope-Energy Dispersive X-ray Characterization

Morphology of CSNP and particle distribution of all samples are presented in Figure-2.

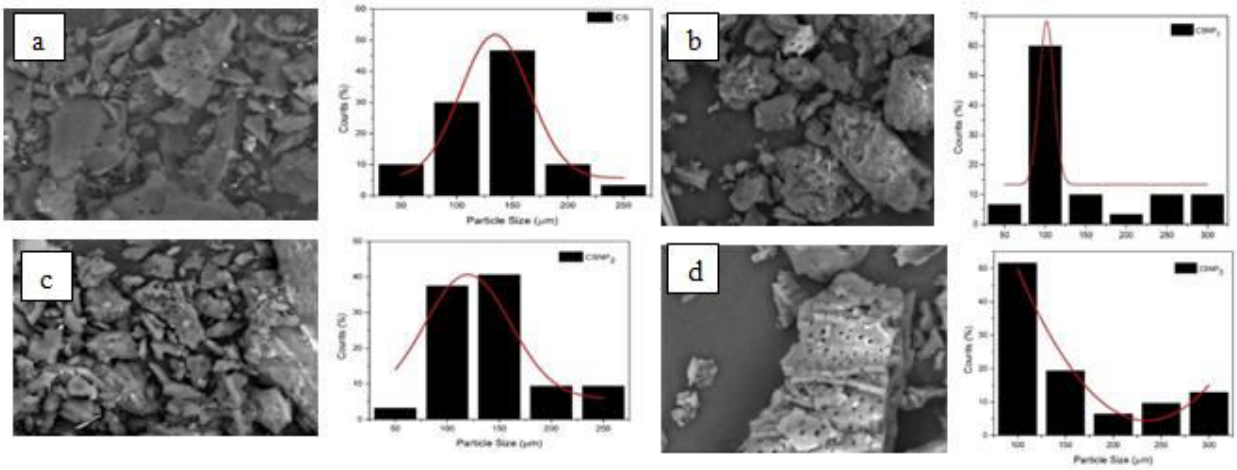

Figure-2. SEM micrographs and Histograms of particle distributions from all samples of coconut shell (a) CS, (b) $CSNP_1$, (c) $CSNP_2$, (d) $CSNP_3$.

The combination of SEM with EDX can identify elements that belong to the phases seen in the microstructural image. The results of the EDX characterization can be seen in Figure-3. The analysis using SEM/EDS shows crystal growth by increasing the particle size distribution. The distribution of the particles is not uniform, and there are pores according to the carbon element. The components that make up charcoal consist of bound carbon, ash, water, nitrogen, and sulfur. Most of the carbon pores are well-formed [28], [29]. From Figure-4d, it can be seen that the pores form large and evenly wide holes. The surface of the pores is clean, not filled with dirt that closes. In addition, Figure-4b shows that the average particle size of all samples is in the range of 50 µm to 300 µm; these conditions good yields for carbon black as mesoporous particles. Electron Dispersive X-ray (EDX) spectrum helps know the elements formed in the sample. The dominant elements contained in the sample are known, and the percentage of carbon elements is used as a reference in determining the phase formed. Based on the results of the EDX test, the main elements in the sample are carbon and oxygen. Other elements such as Ca, Mg, Zr have a lower percentage, below 1%. The highest percentage of carbon was sample CSNP3-elemental composition of CS with EDX analysis presented in Table-3.

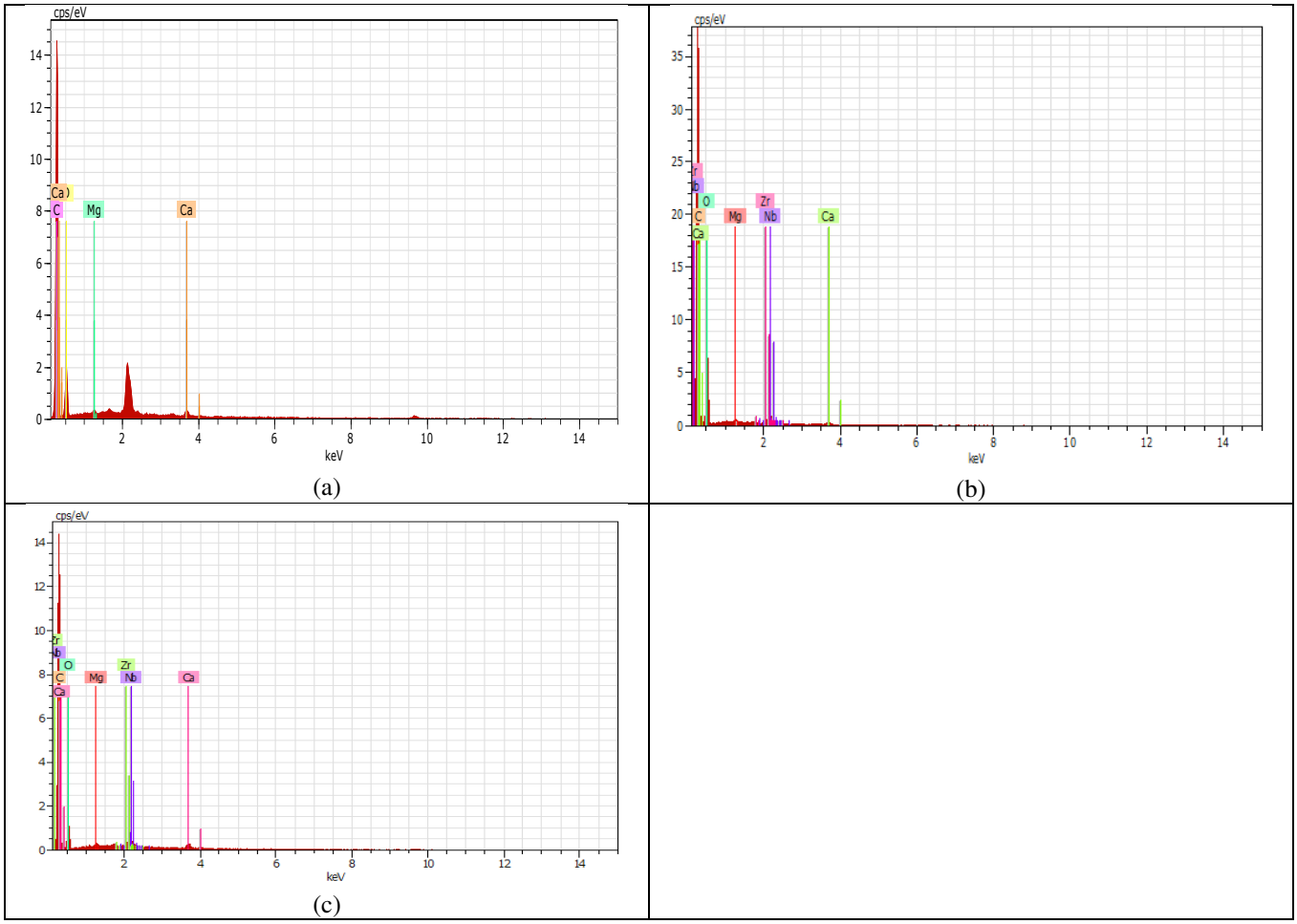

Figure-3. EDX Spectrum of Carbon Black from coconut shell (a) $CSNP_1$, (b) $CSNP_2$, (c) $CSNP_3$.

Based on Figure-3 and Table-3, the EDX analysis of $CSNP_3$ have a more significant carbon content of 76.97 wt%, while $CSNP_2$ is only 69.98 wt% and for $CSNP_1$ 72.28 wt%. It could be due to different concentrations affecting the formation of the carbon [30]. The amount of carbon produced in this study is more significant than in previous studies [31]–[33]. In Table-3, it can also be seen that other elements like O, Ca, Mg, Nb, and Zr, which are the essential ingredients of coconut shells.

Table-3. Elemental composition of CS with EDX analysis.

Element	$CSNP_1$		$CSNP_2$		$CSNP_3$	
	Wt%	At%	Wt %	At%	Wt%	At%
C	72.28	72.12	69.98	77.03	76.97	84.14
O	26.47	21.48	27.03	22.34	17.92	14.71
Ca	1.25	0.40	0.49	0.16	1.62	0.53
Mg	-	-	0.27	0.15	0.31	0.17
Nb	-	-	1.61	0.23	2.16	0.30
Zr	-	-	0.62	0.09	1.03	0.15
Total	100 %		100%		100%	

3.3 Fourier Transform Infrared (FTIR) Characterization

Functional group analysis was performed using the Fourier Transformer Infra-Red (FTIR) model Shimadzu IR-Prestige-21 made in Japan. The resolution used is 4.0. The wavenumber ranges 500 cm^{-1} - 4500 cm^{-1}. The FTIR CS graph is shown in Figure-4.

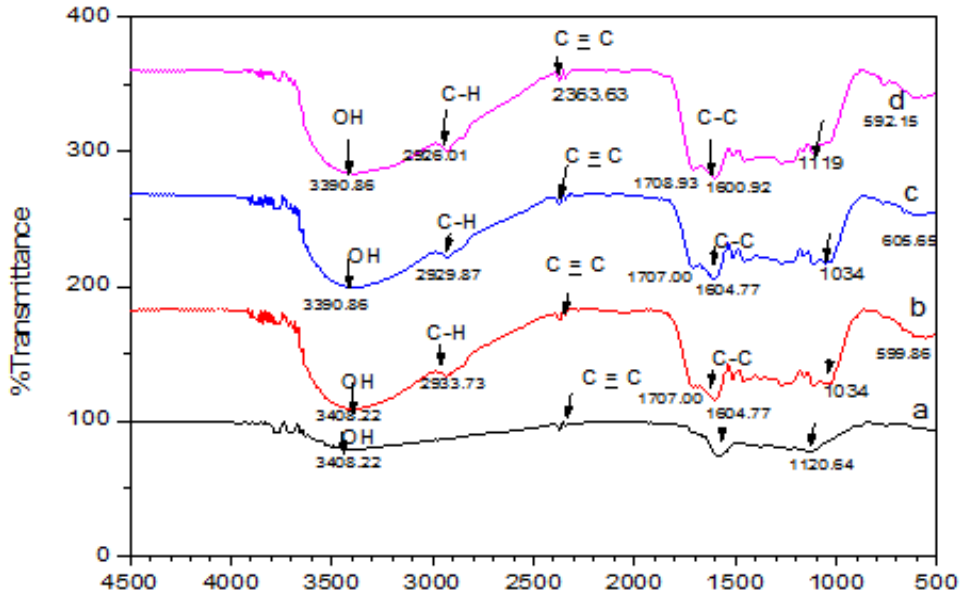

Figure-4. FTIR Spectrum of (a) CS, (b) $CSNP_1$, (c) $CSNP_2$, (d) $CSNP_3$.

The spectrum results show that the absorption peaks of $CSNP_1$, $CSNP_2$, and $CSNP_3$ have the same pattern. $CSNP_1$, $CSNP_2$, and $CSNP_3$ showed a wavenumber of 3390.86 cm-1; this indicates a vibrational strain of the OH group on the surfaces bound to the C element that contained in the charcoal. In Figure-6 (a), (b), (c), the appearance of an absorption band at the peak of 1707.00-178.93 indicates the presence of carbonyl lactone, aliphatic ketones and aldehydes conjugated with the carbon black aromatic structures. It can be said that the synthesis of CS into CB was successfully carried out. The absorption band 2926.01-2933.73 in the synthesized sample shows that there seems to be. This happens because CS is synthesized with HCL solution [34], [35]. In addition, an absorption band appears at 592.15-605.65, which is a C-C stretch band according to the characteristics of the low cellulose composition [36].

3.4 Thermal Characterization

Thermal analysis using Thermal Gravity Analysis (TGA) is presented in Figure-5. The TGA thermogram instrument used is the TA Instrument Model SDT 2960, with a sensitivity of 0.1 g. Air or 99% N_2 as cleaning gas with a flow rate of 100 $mLmin^{-1}$.

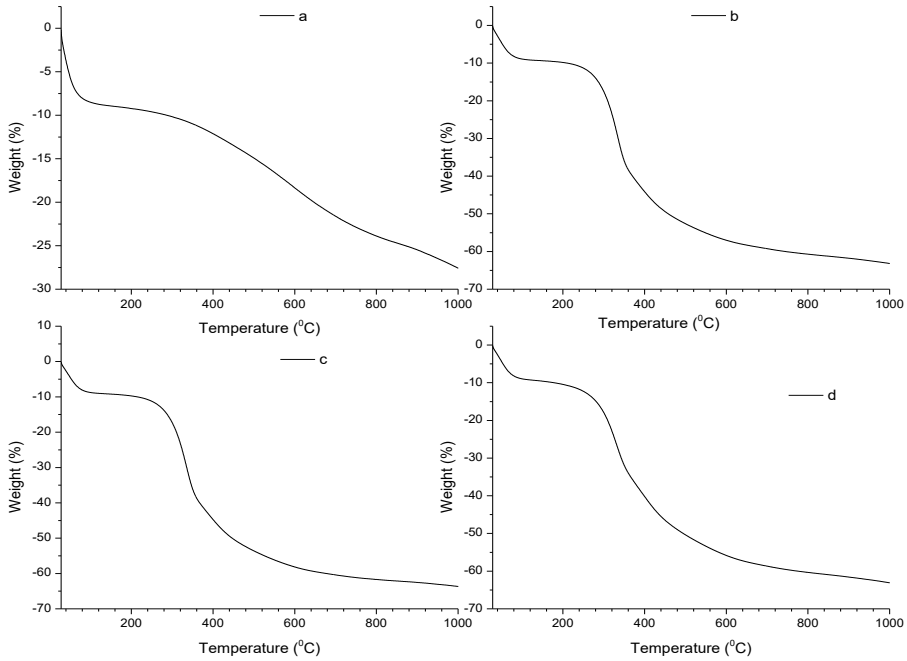

Figure-5. TGA of coconut shell (a) CS, (b) $CSNP_1$, (c) $CSNP_2$, (d) $CSNP_3$.

The decomposition reaction that occurs in the TGA test can be described in Figure-5. Correspondingly, the TGA curve shows an endothermic peak at a temperature of 50-100°C, indicating that the maximum heat is absorbed into the sample. The endothermic peak is associated with phase reduction and decomposition reactions. At a temperature of 100-120°C, evaporation occurs from the water, and up to 27 °C, the cellulose begins to decompose. The distillate contains organic acids and a small amount of methanol. Vinegar is formed at a temperature of 200-270°C. Then at a temperature of 270-310°C, an exothermic reaction occurs by encouraging the breakdown of cellulose into a solution of pyrrolidine, wood gas, and a little tar. Organic acids with low boiling points are formed, such as vinegar and methanol, while wood gas consists of CO and CO_2. Lignin decomposition occurs at a temperature of 310-500°C, more tar is produced while the pyrrolidine solution and CO_2 gas are reduced while CO, CH_4, and H_2 gases are increased. Temperature 500-1000 °C is the stage of refining charcoal or carbon content [37]–[40]. The decrease in mass in CS and CSNP at each stage is shown in Table-4. Figure-5 and Table-4 show that the thermal stability of $CSNP_1$-$CSNP_3$ is more stable than CS.

Table-4. Decreased weight of CS and CSNP

Sample	Stage	T onset (°C)	T end set (°C)	Loss Weight (%)	Δw(%)
CS	1	28	72.6	8	8
	2	95.7	199	9.5	1.5
	3	199.7	1000	27.9	18.4
$CSNP_1$-$CSNP_3$	1	28	72.6	8.5	8.5
	2	72.6	234	10.8	2.3
	3	234	332	29.4	16 .7
	4	332	799	62.1	32.7

Based from results of morphological that the carbon pores ordered to graphite structure on the coconut shell active carbon, which this conditions improved conductivity of the material. While no chemical unsure beside carbon and oxygen, the active carbon was pure and potentially for the electrode of supercapacitor [27, 28]. As good for supercapacitor materials, the prepared activated carbons must showed carbon structure in morphological structure. In this research, carbon structure informed based from Figure-2. This research, the preparation of carbon black nanoparticle from coconut shell will contribute to the impact of environmental friendly and well economical process for candidate super capacitor electrode materials, respectively.

4. CONCLUSIONS

Synthesis of CS by coprecipitation method with various solvents affects the thermal and microstructural properties of the carbon black produced. Morphological results showed that most of the carbon pores were well-formed, and the dominant elements in the sample were carbon and oxygen. The appearance of the absorption band belonging to the CB in the synthesized CS sample was confirmed from the FTIR results. In addition, from XRD analysis, the particle size of $CSNP_1$, $CSNP_2$, $CSNP_3$ samples was (33.2, 52.92, and 54.98) nm with an amorphous structure. The endothermic peak is associated with phase reduction and decomposition reactions. In general, based on the analysis results, it was found that CSNP synthesized by the coprecipitation method had better thermal stability than CS.

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