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Physical, Chemical and Morphology Characteristics of the Malaysian Coconut Shell Powders (CSP)

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Abstract- Coconut shell (CS) raw materials used in this project were obtained from our previous by-products of copra processing. The methodologies involved are (i). Collection of CS materials, cleaned, air dried and kept in a proper container prior to production of coconut shell powder (CSP). CSP is a potential industry material and it has been reported by several industries as natural raw materials for production of repellent fillers, adhesives, composites and other high value added of non-food products. In this research, CS was further processed into powder form (CSP) by several steps of processing that will be discussed further in this finding; and the second methodology - (ii). A detailed evaluation of CSP was conducted for its chemical and physical properties. These analyses covered the determinations of: i. Color profiles; ii. Bulk density; iii. Screening on the morphology of CSP using a Scanning electron microscope (SEM); iv. Evaluation of fiber characterizations and v. Measurement of oil residual in the CSP. The aims of this research are to produce specialty CSP materials, identified the physical and chemical characteristics of the CSP; and highlighted CSP as high potential value-added products for non-food uses as an extra income for coconut farmers and related industries.

Keywords- Coconut shells, coconut shell powder, physical, chemical and non-food products.

I. INTRODUCTION

Coconut shells are widely used in a non-food downstream processing industries to produce charcoal, activated charcoal (AC), shell crafts, kitchen-wares, specialty materials such as material construction and other phytochemical productions. The coconut shells (CS) raw materials can be obtained from by-products of copra processing and coconut milk productions. A CSP is very rigid material and it is used as construction materials and as natural aggregate in the concrete (Venkateswara et al. 2015) and as aggregate application in a light weight concrete making (Afolayan et al. 2017).

Other previous studies mentioned that the CSP as potential bio-composite materials are fabrication of coconut shell fibres-based composites using different matrixes with a cost effective and eco-friendly bio-composites, which affecting the market values of coconut shell (Somashekhar et al. 2018).

Another research showed some potential of using CSP at 10-20% as alternative material for interior parts of aircraft, spacecraft, ships, electronics, and automobiles (Vasu et al. 2017). A study have found an additional of 3% of CSP concrete, shown a better compressive strength as compared to a normal concrete (Leman et al. 2007) and others detailed investigation and finding was done by Shrestha (2016) on chemistry, structure, mineralogy of lignite and coconut shell-based activated carbon fiber (ACF), analyzing using several multiple characterization techniques including x-ray diffraction (XRD), x-ray powder diffraction (XRPD) analysis, field emission scanning electron microscopy (FE-SEM), multiple internal reflectance (MIR) or attenuated transform reflectance and fourier transform infrared spectroscopy (ATR-FTIR), and scanning electron microscopy coupled with energy dispersive x-ray (SEM-EDX).

A CSP is a potential industry material and it has been reported by several industries in Malaysia as raw materials for productions of mosquito's repellent fillers, adhesives, composites and other high value added of the non-food products. In this research, coconut shell (CS) was converted into powder forms to produce coconut shell powder (CSP) by several steps of semi-industrial processing. The observations are focused on the evaluation of CSP for its chemical and physical properties.

In this research, the scope of analysis is covering for the evaluations of –

- Morphology screening of CSP using a Scanning Electron Microscope (FESEM),
- Fibre characterizations of the CSP, and

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- Physio-chemical properties of the CSP. The aims of the researches are to identify the physical and chemical properties of the CSP and highlighted the R&D informative for non-food products as potential value added and as a new income for coconut farmers and industries.

II. MATERIALS AND METHODS

1. Processing of CSP Powder and Selections of the CSP Samples:
The Coconut shells (CS) collected from a copra waste were cleaned, air dried and kept in a proper container prior to produce coconut shell powder (CSP). These coconut shells (CS) were obtained from previous copra processing dried under 60oC, 80oC and solar dryer conditions. The processing of the CS involved of crushing and milling as Fig. 1.

(a) Coconut Shell Samples.

(b) Crushing & Sieving.

(c) A Bulk of CSP Products.
Fig 1. Diagram of Crushing, Milling and Sieving of the CSP Products.

2. Initial CSP Colour characteristics:
The ground samples were evaluated for the basic physical tests of colour. The colour parameters were measured for its L, a and b values by a Chromameter instrument (Minolta). The system was calibrated before used. The CSP samples were weighed for 50g and filled manually in a petri dish, sealed and tapped vertically for 10 times before measuring the colour parameters.

3. A Bulk Density of CSP:
A bulk density was conducted using a glass syringe with a capacity of 10 mL volume. This in-house method was used as a simple methodology by weighing a marked volume of CS, followed by tapping for 10 times in order to compact and achieved a marked volume of 10mL, then a filled and standard volume of CSP was weighed. The bulk density of the CSP was calculated based on the formula (1) below:

A Bulk density =

$$\frac{\text{Weight of filled CSP amount in a marked 10mL bottle}}{\text{Volume of bottle (10mL)}} \quad ......\ (1)$$

4. SEM Morphology of CSP:
A scanning electron microscopy was used to measure a surface morphology of coconut shell (CS) particles and coconut shell powders (CSP) observed under a Field Emission Scanning Electron Microscope (FESEM)– Supra 55VP (Fig. 2) at CRIMLab UKM, Malaysia.

The CS and CSP samples were cut into small pieces and powdered respectively before placing onto the sample stubs. Prior to morphology scanning, the samples were attached onto a sample holder and coated with gold using sputter cutting (SEM Coating System Machine). The morphology of the CSP was observed using a SmartTiff, version V02.01, Copyright © 2008-2008 Carl Zeiss Microscopy Limited.

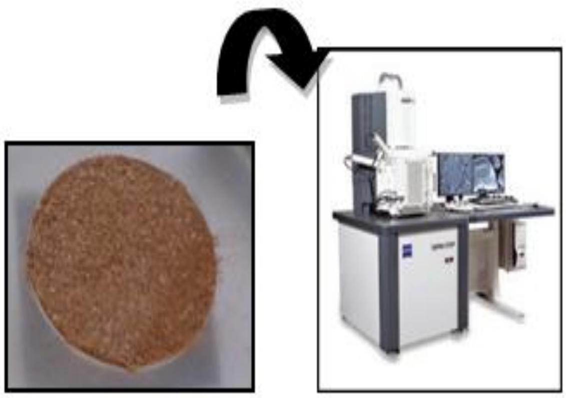

Fig 2. A CSP sample and a Field Emission Scanning Electron Microscope (FESEM) for Surface Morphology Analysis.

5. Fibrous Characteristics:
All the fibrous analyses were done by MARDILab, located at MARDI Headquarters Serdang Selangor, Malaysia. The

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procedures and method of analysis are compiled by MARDILab as standard in-house and accredited methods.

6. Fourier Transform near Infrared (FT-NIR) Profiles of CSP:

A spectroscopy (FT-NIR) was used in order to detect the compound functionality present in a coconut shell-based powdered (CSP). A FTIR spectroscopy studies were carried out by a Fourier Transform near Infrared (FT-NIR) with an Imaging System (Perkin Elmer, model Spectrum 400 FT-IR/NIR). The adsorption in the infrared (IR) region is recorded due to the rotational and vibrational movement of the molecular groups and chemical bond of a molecule (Özacar et al. 2008).

7. Fat Residual in Coconut Shell (CS):

A fat residual analysis was determined with slightly modification using a Velp scientifica; model SER 148 Solvent Extractor, Italy. A 5g CSP was weighed in a cellulose thimble and fix into a solvent reservoir of the system. Petroleum ether at 40-60°C boiling points was used for extracting fat from the CSP. The oil obtained was calculated for residual oil/fat obtained from the CSP samples. The residual of oil/fat is calculated as (2).

$$\textbf{Extract (\%) = Extract x 100 / (Sample)} \quad \textbf{....... (2)}$$

Where:
Extract (g) = (Total - Tare)
Sample= sample weight (g)
Tare= weight of the empty extraction cup (g)
Total= weight of the extraction cup + extract (g)

III. RESULTS AND DISCUSSION

1. CSP Productions from Copra Processing and Its Initial Physical Properties for Obtain the Best CS Samples for Further Physical and Chemical Analyses:

The natural and origin colour of CSP is natural brown. In Fig. 3 showed the CS obtained from copra processing (MH solar, MH 60oC and MH 80^{o}C) at solar dryer, 60^{o}C and 80^{o}C oven treatments respectively. The solar dryer method gave a very low quality, deteriorate and present moldy growth on the copra samples, in addition the CS produce was found contaminated and not suitable for further CSP processing.

In copra processing at 80 ^{o}C, it gave a yellowish and darker copra and it have affected the shell (CS) colour quality. The copra dying at 60^{o}C have given the best quality of white copra and shell (CS), thus this CS was selected for further processing of coconut shell powder CSP samples and selected for our further chemical and physical analyses. The properties of the coconut shell produced were better in colour and appearance as compared to CS dried at solar dryer and 80^{o}C.

As in Fig. 3, the colour of the CSP treated at 60^{o}C have given a natural colour values recorded at L - 44.727, a - 8.107 and b - 23.897. The solar dryer treatment gave more lighter in colour (L 46.870) but slightly moist, and the 80oC drying treatment resulted in more dried and heated, that have changed the natural CS colour and oily properties due to their colour was too darker as compared to 60oC CSP.

The Solar dryer treatment presenting less whitish at L-43.790, and a darker appearance in a and b values. The crushed and grinded CSP sample colours are shown in Fig. 3 below. According to Kumar et al. 2013, their coconut shell powder is a light brown material which has a bulk density of 0.7gcm3. Thus, the CSP treated at 60oC was chosen as raw materials for the next CSP analysis, as due to its dry appearance, natural dried shell colour and this treatment also produce a high copra quality in white appearance and suitable for further VCO production.

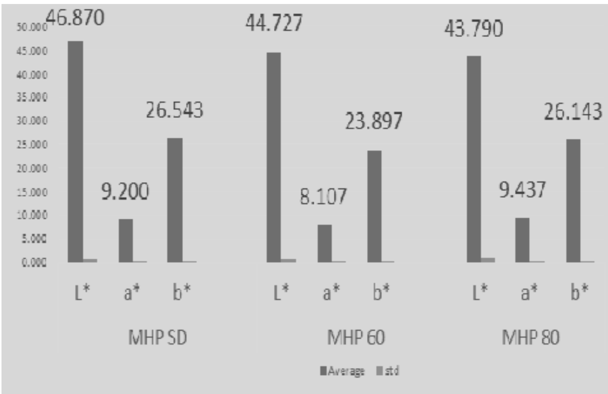

Fig 3. Coconut Shell Colour Characteristics Obtained from Different Copra Making Heat Processing.

Table 1. A fine CSP yield obtained at 72.079 % and its by-product was at 16.098% at an initial grinding. Other larger granulated CSP obtained at approximately at 11.823% (balance).

Table 1. Recovery of CSP During Initial Grinding.

CSP powder (%)	CSP waste (%)	Granulated CSP (larger particles)
72.079 ± 10.249	16.098 ± 9.689	~ 11.823%

2. A Bulk Density of CSP:

In Table 2 below, the bulk density of the CSP were recorded of 0.53, 0.65 and 0.55 g/mL for SP, 1.0mm CSP and 0.5 mm CSP sizes respectively. The bulk density of the CSP produced were lighter than the commercial produce this may due to the shell obtained from copra processing and contained slightly oily materials (see Table

4). From literature, Coconut shell powder was reported as a source from a local coconut miller and the CS has a bulk density of 0.7gcm-3 (Kumar et al. 2013). As a comparison a research done by (Afolayan et al., 2017), a machine crushed coconut shell aggregate gave the highest percentage of CS aggregate between size 14-20 mm.

According to Agunsoye et al. (2014), their processing of the coconut shell was starting from the unprocessed coconut shell followed by sun dried, ground and sieved into different sizes. The CSP produced in this research are able to use as different grade of CS powdered materials by crushing at difference sieve as in Table 1.

According to Kumar et al. 2013, because of the increasing production of agricultural waste, many researchers have attempted to use coconut shells as filler material in the concrete and the previous studies reported coconut shells as a filler material and a light weight concrete can be produced. The coconut shell powder (CSP) has also reported as a complex structure.

Table 2. Bulk Density and size of CSP Samples.

CS Materials	Bulk Density (g/mL)	Size
CS (ref [1])	0.70	-
CS Aggregates (ref [2])	-	14-20 mm
CS particles	1.60	200-800μm
SP Materials	0.53	3-5 mm
1.0 mm CSP	0.65	1.0 mm
0.5 mm CSP	0.55	0.5 mm

Ref:
1. Kumar et al 2013;
2. Afolayan et al 2017; and
3. Bhaskar et al. 2013

3. Field Emission Scanning Electron Microscopy (FESEM) Of CSP at Two Coconut Plantations:
A crushed and cut sites of the coconut shells (CS) illustrated a shiny appearance and rigid surface due to its high silica content. The site of coir base on the CS was clearly observed with an average of 945.64 nm and 1100.83 nm (Table 3). The CS holes suspended for its coir on the coconut shells showed the CS from MARDI Bagan Datuk (MHP) was larger than the CS from MARDI Kluang (MK) for the MATAG coconut variety.

Table 3. Fibrous Hole Diameter (nm) on the CSP Samples Obtained from MARDI Bagan Datuk and MARDI Kluang under FESEM instrumentation.

Coconut shell MARDI Kluang (nm)	Coconut shell MARDI Bagan Datuk (nm)
945.64 ± 197.49	1100.83 ± 336.39

The averages of 7 replicate samples measured.

The CSP particles are course and harden powder, in Fig. 4 and 5 below have illustrated the shape of a particle of CSP with 3.771 mm (Figure 4) and 3.393 mm (Figure 5) a like stone and having rigid materials. The course particles showed a very cracked surface due to the CS materials was very rigid and strong due to its silica content. The particles are brownish and shiny at the cut or crushed surface.

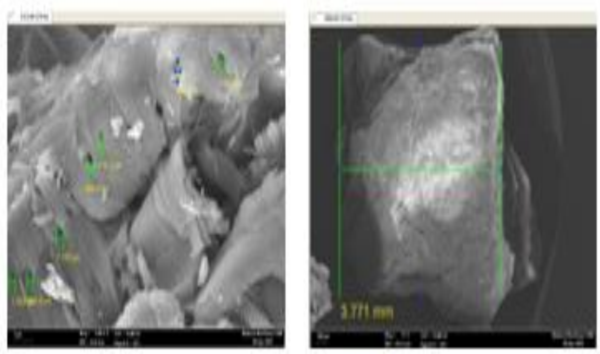

Fig 4. MARDI Kluang Coconut Shell (CS) Characteristics.

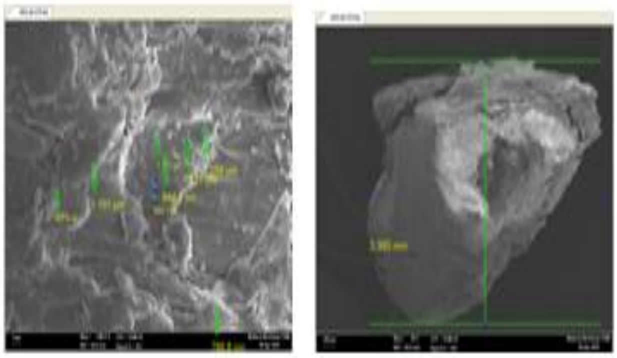

Fig 5. MARDI Bagan Datuk Coconut Shell (CS) Characteristics.

4. Characteristic of CSP Fibre Properties:
In Figure 6, in soluble and crude fibre of CSP achieved up to 88.08% and 52.18%, respectively, this means it is more than half of the shell compositions are bonded with rigid fibrous materials. After an acid treatment, the CSP was increased up to 74.25% and by using a neutral detergent treatment it gave higher fibre portion, which was up to 89.7% of the fibrous properties.

The chemical analyses done by Agunsoye et al. 2014, shows the composition of substances of lignin 29.2%, pentosans 27.8%, cellulose 26.5%, moisture 7.8%. Solvent extractives 4.4%, uronic anhydrides 3.6% and ash 0.7%.

Other elemental analysis of this CSP material presenting carbon 53.88%, hydrogen 6.56%, oxygen 38.56%, nitrogen 0.97% and sulphur 0.03%. The lignin in the studied sample was higher (36.88%) than the lignin of the CSP mentioned above.

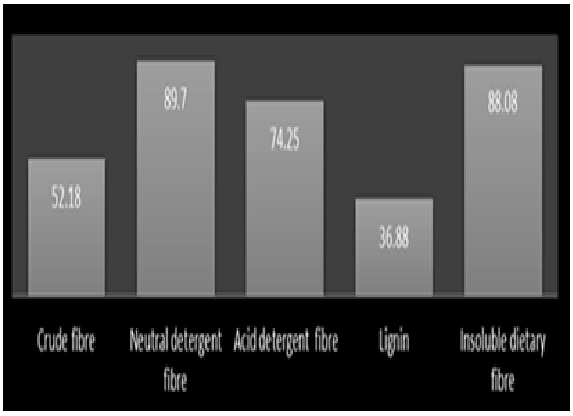

Fig 6. Characteristics CSP Fibre Properties.

5. Fourier Transform Infrared Spectroscopy (ATR-FTIR) Profiles of CSP:

The CS sample was used for determined its FTIR profile. The CSP sample was initially passed through a grinder and crushing at 0.5 mm. The sample was illustrated in Figure 7.

Fig 7.The CSP sample used for FTIR analysis.

A FTIR spectrum of the raw coconut shell is shown in Figure. 8. From the results, it illustrated that a band shifting around the broad peak at 3340.37 cm−1 indicates the possible involvement of hydroxyl group. The peak at 2912.69 cm−1 is due to the CH stretching that cause vibrations of CH, CH2, and CH3 groups. The absorption bands at about 1726.02 and 1238.50 cm−1 show the characteristic of C═C bonds in aromatic rings. The C–O carboxyl bands at 1605.50 and 1252.23 cm−1.

The major deviations are in the regions from 3340.77 and 2912.66 cm−1, which are assigned to the vibrations of N–H and O–H functional groups. The peaks at 1161.45 and 1032.50 cm−1 are attributed to Si–O stretching and bending, indicating the presence of silica. The reference of the coconut shell powder (CSP) FTIR was referred to Zainab et al. 2015.

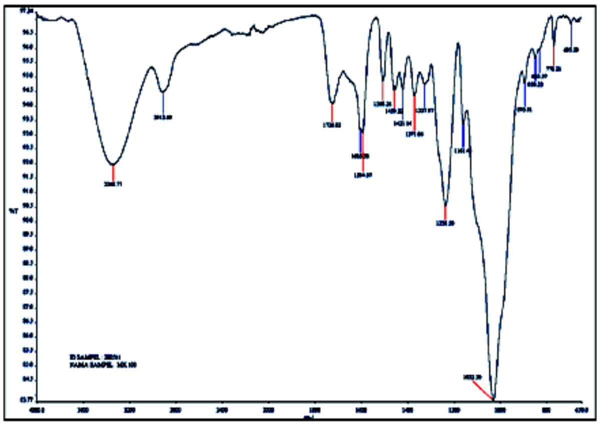

Fig 8. A FTIR of Coconut Shell Powder (CSP).

6. Residual of Oil Content in CSP:

In Table 4, the CS of Copra by products gave a slightly residual of fat content at 0.108, 0.12 and 0.10 % for treatment of 60, 80 and 100oC respectively. This fat residual can preserve CSP from mouldy contaminations due to coconut oil is a natural preservative. At temperature of 35-45 oC and fluctuated condition of solar dryer has given low rate of drying properties, thus there was no residual of fat migrated into the CS sample.

The CS was slightly mouldy after a few days, and it also produce a moist copra due to the inconsistent and fluctuated temperature conditions of the solar dryer, finally produced contaminated copra samples, and the coconut shells (CS) could not be used in this experiment.

Table 4. The CSP Oil Residual by different treatments.

Coconut Shell + copra drying condition (^{o}C)	Oil Residual in the Coconut Shells (%)
35-45 ^{o}C (Solar dryer)	n.d
60 ^{o}C (Oven)	0.108
80 ^{o}C (Oven)	0.120
100 ^{o}C (Oven)	0.101

IV. CONCLUSION

CSP is a natural rigid ingredient, containing silica portion and other compounds. It's a rigid pallet with sandy and stone appearances. This material is insoluble fibrous and having powerful strengthened materials that can be used as a rigid industry material.

A light density of the CSP powder is suitable for carbon bricked formulations as a fuel source, activated charcoal and other premix-blending products such as light concretes and industry materials. These full informative properties of the CSP can be used as a reference for converting agricultural waste into a wealth; and producing value added industry materials.

V. ACKNOWLEDGMENT

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