Journal of Materials Science and Chemical Engineering, 2018, 6, 1-18
http://www.scirp.org/journal/msce
ISSN Online: 2327-6053
ISSN Print: 2327-6045

Physicochemical and Thermal Characterization of Dura Palm Kernel Powder as a Load for Polymers: Case of Polyvinyl Chloride

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How to cite this paper: Djomi, R., Meva'a, L.J.R., Nganhou, J., Mbobda, G., Njom, A.E., Bampel, Y.D.M. and Tchinda, J.-B.S. (2018) Physicochemical and Thermal Characterization of Dura Palm Kernel Powder as a Load for Polymers: Case of Polyvinyl Chloride. *Journal of Materials Science and Chemical Engineering*, **6**, 1-18.
https://doi.org/10.4236/msce.2018.66001

Received: May 10, 2018
Accepted: June 18, 2018
Published: June 21, 2018

Abstract

This work presents the physical and thermal characterization of the dura palm kernel powder of Cameroon for their use as fillers for polymers composites. The powders of palm kernel were obtained using a percussion grinder mill with an industrial microniser which allowed obtaining a powder less than 50 μm with an apparent density between 0,505 ≤ ρ ≤ 0,680 g/cm^3 at 1.56 of relative humidity. The infrared of the powder of palm kernel shows the presence of phenols groups with a large band around 3341 cm^{-1}, -C-H at 2917.02 cm^{-1}and -C-O at 1040 cm^{-1} as the main peaks. The polyvinyl chloride of infrared obtained shows the presence of -C-Cl, -CH_2 and CH as the mains peaks. The infrared of 12.5% of palm kernel powder with polyvinyl chloride shows an increase of the CH_2 and CH bonds and a decrease of the -OH bonds. Thermogravimetric analysis and differential scanning calorimetric analysis of powders, polyvinyl chloride and mixture showed that the mixing powders are intermediate between the polyvinyl chloride and palm kernel powder. The powder decreased the phase temperatures of the mixture from 98.58˚C to 95˚C for the glass transition temperature and from 515˚C to 459˚C for the crystallization temperature. The thermogravimetric curves of palm kernel powder and polyvinyl chloride have showed that these materials lose their different masses in three different phases, and the one of composite (mixture of polyvinyl chloride with 12.5% of palm kernel powder) in two different phases.

Keywords

Dura Palm Kernel Shell, Load for Polymers, Thermogravimetric Analysis, DSC

1. Introduction

Polyvinyl chloride (PVC) is one of the most important plastics used in the world today. They are mostly found in the packaging, electricity, electronics, electromechanical and building sectors [1]. Plastic offers a very great advantage especially in the possibility of its mixture with many other materials at the time of its manufacture [1] [2]. This feature makes the plastic material a financially accessible material for all. Thus, plastic production industries use carbides, glass fibers, carbon fibers [3] [4] [5], especially calcium carbides, as a filler to soften the costs of plastics [3]. Calcium carbide is a mineral material so they are found in large quantities and cheaper on the market. The major disadvantage of such plastic is that they are not biodegradable. Given their importance in the society, the need to replace them either in the form of a load or a form of reinforcement to facilitate its degradation at the end of its life is important. That's why since the 1980s, researchers have been trying to study the possibilities of incorporating plants into polymers during the manufacture of plastics, hence the name Wood Plastic Composites (WPC). In this way, several tests have been successful in the use of talc mineral [6], wood [7] [8], cactus [9], date palm fiber [10] in the form of fibers. The most striking results are the easy formatting (packaging, electricity, electronics...), the lightness of the plastic material (manufacturing, aeronautics...), and treatment (buildings...), next to other properties that are no longer to enumerate [11]. The advantages of the reinforced synthetic polymers with natural fibers are many such as: availability, low costs and abundance of natural fibers [12]. The reinforced plastics material obtained are inexpensive, renewable and nontoxic [5]. The characteristics of different natural fibers have been already described in literature except the one of palm kernel shell [13]. Apart from the charges mentioned above, we note that dura palm kernel powders can also be used as vegetable filler.

The palm kernel shell dura comes from palm oil, plant of the family ARECACEAE (palmae) of its scientific name *Elaeis guinensis* Jacq, referenced by comparison to the botanical collection of TAMAKI MARUHASHI of the national herbarium of Cameroon. This variety of palm oil is less exploited because of its low yield of oils that is the peculiarity of its exploitation [14]. They are found in very large quantities and in more than half of the Cameroon territory, but not maintained. Palm kernel husks represent the part of this least used plant (if so rarely for cooking) and are considered as harmful waste. Like its palm, they are found in very large quantities and much more thrown into nature.

It should be added that the palm oil sector in the world [15] in general and in Africa in particular is in the midst of valorization [16]. For example, Planet reports in "History of oil palm exploitation in Africa", GRAIN, 22 September 2014, in Guinea, palm oil exploitation is a source of stable employment, mitigates rural exodus and develops the local economic fabric. In West Africa, Benin, Côte d'Ivoire and Guinea, artisanal extraction of palm oil is done by the palm producer. Revenues enable them to support themselves and reflect the

wealth of a clan or family. In the Lower Congo River, local knowledge around the palm oil is a rich heritage to be valued also for the protection of biodiversity [17]. In all these countries, the usefulness of palms is limited to oils. The hulls of palm kernel also remain in all these countries, the part of the tree which is not used [4].

Palm kernel husks have been investigated in its use as fuel for cupolas [15] and for the production of activated carbon [17]. The vegetable loads are in full success in its elaboration [18].

Palm kernel shells are available not only in Cameroon, but also in abundance in Africa, the Mediterranean and in Southeast Asia [15]. They are an integral part of the list of oil palm pests [14]. The exploitation of palm kernel husks as an industrial load for polymers economically and socially leads to the valorization of the oil palm agricultural sector and the creation of new enterprises, in terms of the environment, the safeguarding of the ecosystem and scientifically, additional knowledge in the field of plant loads for the development of plastics.

The present study aims to set up a practical, experimental methodology to transformed nut shells into powder. Characterize the powder obtained and then use it as fillers for polymers in the production of plastics. It should also be noted that in the literature we have read so far no work of this kind on the use of hulls of palm kernels as fillers has been realized.

2. Materials and Methods

2.1. Materials

2.1.1. The Raw Palm Kernels Shell of Dura

The hulls of dura palm kernel were collected in Cameroon, in the littoral region and Moungo department. They were identified by comparison with the botanical collection of TAMAKI MARUHASHI N˚103 recorded in the national herbarium of Cameroon under N˚47794/HNC. These hulls come from the palmoil of the family ARECACEAE. Its scientific name is *Elaeis guinensis* Jacq. Determavit TADJOUTEU Fulbert HNC on 04/05/2018. The **Figure 1** below shows the raw palm kernel shell of dura.

The raw palm kernel shells were previously dried until constant mass. The hulls were first crushed using a Retsch brand mill, type SN 100 of series No. 82508001 [19]. The machine was not able to crush them. We looked for the cause and we got that the hulls were very hard. Due to the hardness of the hulls, that was not crushed and ground crush by conventional machine, the design and manufacturing of an impact crusher in NASE Yaounde, with the characteristics listed in **Table 1** was made.

2.1.2. Characteristic Parameters of the Machine

1) Thermogravimetric analysis

Thermogravimetric coupled with differential scanning calorimetry analysis was performed using a LINSEIS branded device connected to a computer with

Table 1. Characteristic parameters of the machine used.

reference mark	Designation	observations
1	Base of the machine	Flat iron of (30 × 20 × 6) mm
2	Three-phase asynchronous electric motor	7.5 HP
3	Inlet Hopper Hull	Steel sheet 1.5mm thick; capacity 20 litres
4	Crusher	Z 30 CDNW 6 - 4 - 2
5	Grinding chamber	Blue sheet 12 mm thick
6	Mechanical sieve for crushing	Blue sheet thickness 3mm, opening holes 1.5 mm hole
7	Mechanical sieve for grinding	Blue sheet thickness 2 mm, hole opening 0.5 mm
8	Crushing efficiency	61 kg/hour
9	Grinding yield	47.25 kg/hour
10	Control of the machine	Electric box secured with automatic stop during an overload and an increase in temperature of the engine.

Figure 1. Raw palm kernel shell of dura.

embedded software for data acquisition. A 150 mg capacity aluminum oxide crucibles were used with a; the speed of the measurement is variable and the combustion gases are nitrogen.

PVC that used was collected from the company DANSUK INDUSTRIAL CO., LTD under the name Vinova and batch number S6830 [20]. This PVC was used as given without any other purification.

2) Fourier transformed infrared analysis

Fourier transform infrared spectroscopy (FT-IR) was performed using the Nicolet iS5 IR spectrometer.

2.2 Experimental Methods

Processing of Raw Palm Kernel Shell into Palm Kernel Powder

Figure 2 shows the process of converting raw dried dura palm husks into powder.

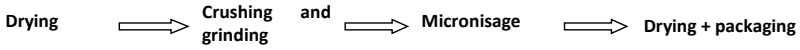


Figure 2. Synoptic diagram of the process of transformation of raw palm kernel shell into powder.

The dried palm kernel shells were poured into the hopper of the mill and are conveyed into the crushing chamber through the opening of the hatch. These are sucked by the centrifugal effect of the rotational movement of the mobile crusher which projects them on the walls of the grinding chamber. The centripetal force (Equation (1)) of the mobile crusher projects the hulls onto the stationary mills of the machine thus providing the necessary energy for their fragmentation.

$$F = m\omega^2 R \tag{1}$$

The crushed hulls pass through the mesh size 1.5 mechanical sieves and are recovered. Subsequently, a sieve of 0.5 mesh is mounted in place of the previous to ensure the crushing of kernel shell previously crushed. The milled powder is transported to a micronizer which refines the powder and gives the desired grade (50 μm). An oven heated to 103˚C for 24 hours allows dehumidification. The appropriately dried powder is immediately stored in plastic bags.

2.3. Characterization of Kernel Shell Powder

Physical Characterization

1) Density

Apparent density: the method described by Ernesto de la Torre Chauvin in 2015 [21] was used to evaluate the apparent density (Equation (2)). The measure of apparent density consists to place an empty volumeter of capacity 1 m^3 on the SEDITECH balance with a precision of 1/1000 and tare. Fill the volumeter with palm kernel shell powder until 1 m^3. For a volume v_i of the powder, record the mass m_i. Determine the value of the mean and the standard deviation which represents the density ρ_v of the micronized palm kernel shell powder.

$$\rho_v = \frac{m_i}{v_i} \tag{2}$$

where m_i = mass of the powder and v_i = volume of the powder.

Wetted density: the method described by Ernesto de la Torre Chauvin in 2015 [21] was used to evaluate the wetted density. It consists to take an empty flask of volume V_0 and weight the mass of the flask (m_0). Add a mass m_1 of water, and a mass m_2 of powder in the flask and let stand. Let all the mixture stand avoiding the formation of air bubbles in flask and note the mass (m_3). The wet density ρ_m is given by Equation (3). Calculate the average and the standard deviation of the records.

$$\rho_m = \frac{\rho_{\text{water}}\left(m_2 - m_0\right)}{\left(m_2 - m_0\right) - \left(m_3 - m_1\right)} \tag{3}$$

2) Granulometry of micronized palm kernel shell powders

The particle size is sieved using an Afnor 50 μm aperture calibrated sieve in order to ensure the wear of the microniser jaws.

3) Moisture content of powder

To evaluate the moisture contained of powder, we used the method described by Dietsch *et al.*, 2014 [19]. Six ceramic crucibles containing the kernel nut shell powder whose mass m_h was previously measured (with a mass of the crucible and the powder) were deposited for in an oven at 103˚C until constant mass were obtained. The dried masses (m_d) were recorded. The moisture content (MC) of each sample is obtained by Equation (4) below.

The moisture content (MC) was calculated using the Equation (4) below.

$$MC = \frac{m_h - m_d}{m_d} \times 100 \tag{4}$$

where m_h is the wet mass of powder, and m_d the anhydrous mass of the powder

4) Thermal characterizations

The thermal behavior of the shell powder is obtained by interpretation of the TG and DSC thermograms performed by the LINSEI instrument of the Physico-chemistry of Mineral Materials Laboratory, Faculty of Science, University of Yaounde 1—Cameroon. The heating rate is 10˚/min. The flue gas is oxygen. The crucible is alumina oxide of capacity 150 mg; the loading mass is 100mg; the initial heating temperature is 20˚C. We were given a thermogram TG coupled DSC with data records.

5) Fourier transformed infrared analysis

The FTIR analyses were carried out using the iS5 spectrometer. The spectra were acquired in the range of 4000 to 480 cm^{-1} at a resolution of 4 cm^{-1}. Characteristic absorptions bands of the processed composites were registered.

6) PVC

The analyses performed on the hulls were also made on PVC under similar conditions.

7) PVC mixture with 12.54% of palm kernel shell powder before shaping.

According to literature, mixing powders (PVC + filler + additives) have to go through analysis before shaping [22] [23]. The most used analysis in companies is thermogravimetry and differential scanning calorimetry analysis in order to ensure a good mixing and success in the shaping of the load with the polymer (PVC) and its additives [2] [23] [24]. A dosage of 12.54% load of kernel nut shell powder was chosen. This dosage is chosen because the calcium carbide feeds frequently used in companies in Cameroon do not exceed 20%.

A mixture of 33.33 kg (81.17%) of PVC, 1.30 kg (3.17%) of stabilizer, 0.70 kg (1.70%) of lubricant, 0.48 kg (1.16%) of plasticizer, 0.09 kg (0.23%) of titanium dioxide, 0.01 kg (0.03%) of black charcoal, 5.15 kg (12.54%) of hull powder, corresponding total of 41.07 kg equivalent to 100% of mixture. The different percentages of the additives were chosen according to the standard applicable in the Cameroon company. The mixture is put in a mixer (steaming of the mixture) to make them perfectly homogeneous. A sample is taken and a thermogravimetric

and differential scanning calorimetry analysis is done with the same apparatus as described before and under the same conditions.

Apart from the analyses of thermogravimetric and differential scanning calorimetry, all the manipulations were done 6 times and the means of the six tests were expressed with standard deviation.

3. Results and Discussions

3.1. Transformation of Raw Palm Kernel Shell to Palm Kernel Powder

The passage of palm kernel shell in a crusher-mill and then in an industrial micronizer helped to transform palm kernel shell of dura palm into palm kernel powder with a size smaller than 50 µm [3] [6] [7]. After micronization, we sieved using a 50 µm afnor sieve. Analysis of the obtained poweder after sieving, shown that nothing left in the sieve used. This allows confirming the particle size of 50 µm was obtained with the manufactured crusher-mill-micronizer. This may be comparable to the granulometry of the calcium carbide used as the filler in PVC.

3.2. Apparent Density

Density is an important parameter for plastic composite in view to obtained material that is less heavy. The average values obtained in the case of apparent density is: $\rho_v = 0.680 \mp 0.013 \mathrm{g/cm^3}$. This proves that there is not enough vacuum between the powder seeds. This density can be compared to the density of plant fibers such as bagasse [25], oil palm empty fruit bunches [26] [27] [28]. The result can be comparable to those obtained by Ernesto de la Torre Chauvin [21].

3.3. Wet Density

The average values obtained in the case of wet density is:
$\rho_m = 0.505 \mp 0.006 \mathrm{g/cm^3}$. This result proves that the shell powder of dura is hydrophilic probably due the presence of hydroxyls group that can fix water molecules via the formation of hydrogen bounds. The real density ρ_r of the palm kernel powder shell is between the two densities obtained $0.505 \le \rho_r \le 0.681 \mathrm{g/cm^3}$.

When measuring masses for mixing (steaming), the masses are measured poured into breasts. During shaping, the resin melts and the powder absorbs the resin to crystallize the materials. The results obtained, allow saying that the density of the kernel powder from Cameroon is between 0.505 and 0.680 g/cm^3. In literature, it is found that the density of some polymers is: 1. 38 to 1. 41 for PVC), 0.89 to 0.93 for PE, of 0.85 to 0.92 for PP and 1.12 to 1.16 g/cm^3 for Nylon 6.6. The values of the density of palm kernel powder obtained, compared to the density values of some plant fibers used to reinforced polymers such as hemp (1.5 g/cm^3), feather (0.9 g/cm^3), Wool (1.3 g/cm^3), sisal (1.3 - 1.5 g/cm^3) [12], show that the plastic obtained after processing will be very light. This offers a very great advantage to palm kernel powder as filler for polymers, especially with

a high loading percentage, now that the automotive and aeronautics sectors are making the construction lighter to limit greenhouse gas emissions.

On the other hand, the hydrophobic behavior of the shell powder remains unchanged after shaping. As a result, the rate of absorption and desorption of fluids is washed away [6]. Vegetable materials are degradable. These results show that the degradation of the plastic obtained will be accelerated at a high load rate. These phenomena make it possible to look at palm kernel husk powder as filler for polymers.

3.4. Moisture Content of Shell Powder

The means value obtained from these results showed the moisture content of palm kernel powder is 1.6% ± 0.1%. From this moisture content value, it appears that the shell powder does not have enough water for hydration and that this measurement was made when the powder had just been dried to be ground. It will not be doubtful to find such a high humidity level, as the result brought by Ernesto de la torrechauvin in his thesis [21]. Comparing the result obtained with the result obtained by Ernesto de la torrechauvin it can be concluded that, the palm kernel powder was taken well dried.

3.5. Infrared Fourier Transform Analyses

On Figure 3 below, we presented the infrared of raw palm kernel powder, raw PVC and of composite (PVC + 12.5% of palm kernel powder).

In Figure 3, FT-IR analysis of raw palm oil kernel shell shows different peak intensities. The large peak intensity at 3363 cm^{-1} can be attributed to O-H stretching group of alcohols (cellulose content in raw palm oil kernel shell). The intensity at 2950 cm^{-1} can be attributed to C-H stretching group of alkanes or the vibrations of the methoxy group of lignin. The intensity peak between 1718 - 1700 cm^{-1} can be attributed to C = O stretching, or to C-C stretching of aromatic

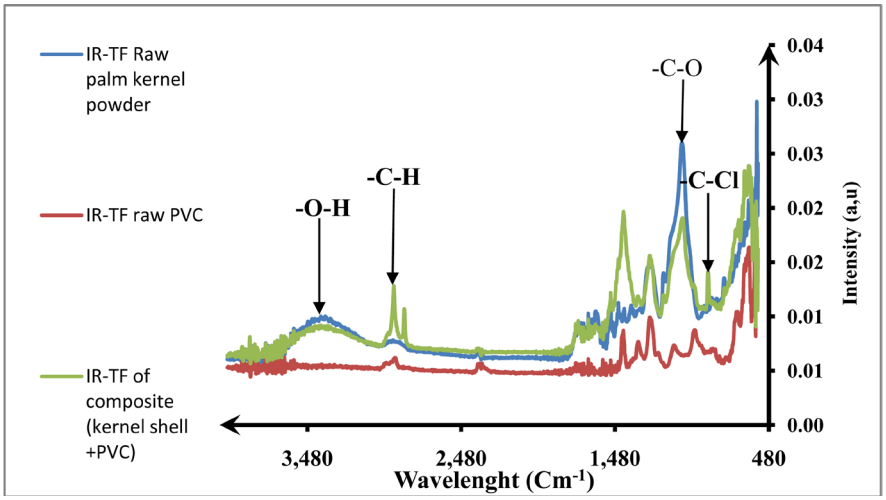


Figure 3. Infrared of raw palm kernel powder, raw PVC and composite (PVC + 12.5% of palm kernel powder).

ring or carboxyl group of lignin. The peak between 1300 - 1000 cm^{-1} and those of 1243 - 1044 cm^{-1} could be attributed to C-O stretch of alcohols, esters or ethers. These results are in agreement with data found in literature concerning palm kernel shell. Kundu *et al.* 2015 [12], Hidayu *et al.* 2013 [2] obtained the same shape and result during the studies of palm oil kernel to produced activate carbon.

The FT-IR analysis of polyvinyl chloride shows peak intensities at 2909, 1425, 1325, 1253, 1095, 962,825 and 607 cm^{-1}. The peak intensity at 2909 cm^{-1} can be attributed to the vibration of C-H. The intensity at 1425 cm^{-1} can be attributed to -C-H deformation. The intensity at 1325 cm^{-1} can be attributed to $-CH_2$ deformation. The intensity at 1253 cm^{-1} can be attributed to -C-H rocking mode or out of plane angular deformation of -C-H. The intensity at 962 cm^{-1} can be attributed to trans -C-H wagging mode or out of plane trans deformation. The intensity at 825 cm^{-1} can be attributed to -C-Cl bond stretching. The intensity at 607 cm^{-1} can be attributed to -CH cis wagging mode. These peak intensities are in accordance with data found in literature concerning PVC [2] [4] [16] [29].

The FT-IR analysis of polyvinyl chloride with 12.54 palm oil kernel mixture shows different intensities peak. The peak intensity at 3301.52 cm^{-1} can be attributed to the vibration of -O-H. The intensity at 2917.02 and 2849.13 cm^{-1} can be attributed to -C-H. The intensity at 1424.71 cm^{-1} can be attributed to -C-H rocking mode or out of plane angular deformation of -C-H. The intensity at 1424.71 cm^{-1} can be attributed to trans -C-H wagging mode or out of plane trans deformation. The intensity at 1041.05 cm^{-1} can be attributed to -C-O. The intensity at 874.56 cm^{-1} can be attributed to C-Cl. We can note a decrease in intensity at 3301 cm^{-1} of the -O-H groups of the palm oil kernel which can means that there is a new bond that was create between the PVC and palm oil kernel. The same observation was done at 1041 cm^{-1} with the increasing intensity compared to PVC and decreased compared to palm oil kernel. At 2917, 1424, 1253, we observe a strong increase of intensity of the groupings. All these increases, decreases and creations of grouping may imply an interaction between palm kernel nuts powder with PVC [2] [24].

3.6. Thermogravimetric and Differential Scanning Calorimetry

In **Figure 4**, we present the thermogravimetric and differential scanning calorimetry of raw kernel nut shell powder.

Figure 4 shows the mass loss (TG) (in red) and the differential scanning calorimetry (DSC) in blue of the raw kernel nut, for a temperature velocity of 10˚C/min at atmosphere controlled by oxygen.

In this figure, it appears that the raw material exhibits three degradation phases:

1) Dehydration: the degradation starts at 101˚C and leads to about 1.13% of mass loss. This phase corresponds to linked water evaporating from the material [6] [7].

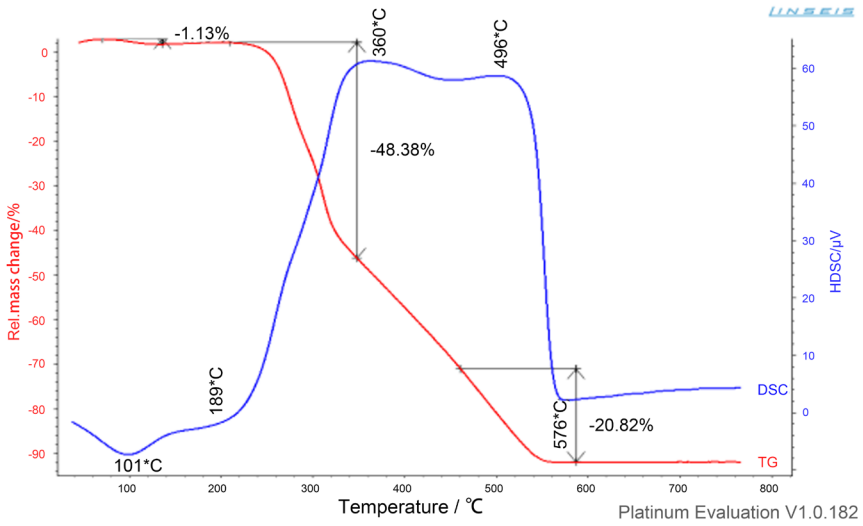


Figure 4. Thermogravimetric and differential scanning calorimetry of raw palm kernel shell powder.

2) Hemicellulose and Cellulose degradation: the degradation starts at 189˚C, end around 380˚C and lead to about 48.38% of mass loss. In this phase hemicellulose decompose between 189˚C and 300˚C, the cellulose decompose between 300˚C and 380˚C. When the two polymers are degraded, the material re-equilibrates by forming others phases and releasing a calcinate.

3) Lignin degradation: the degradation starts around 400˚C and leads to about 20.82% of mass loss. The entire bonds are broken, a large part of the material is pyrolyzed and only the ashes remain.

These results are in agreements with the results obtained by others researchers [24].

The DSC shows an endothermic peak of heat at 101˚C. During the degradation of cellulose and hemicelluloses, DSC presents a peak at 360˚C and during the degradation of lignin there is another peak at 496˚C. It can be also note that the ignition temperature is 200˚C and the burnout temperature is 580˚C for the raw palm kernel shell.

As the aim of this work is to use the palm kernel shell as filler, it is necessary to study the behavior of palm kernel shell between 0 and 250˚C. The ideal processing temperatures of several polymers are between 80˚C for polyethylene and 250˚C for polytetrafluoroethylene or between 170˚C and 205˚C for rigid PVC that we studied [2]. These analyses are made in order to understand the phenomenon of polymer degradation (PVC) and that of the loads (palm kernel shell) in the interval frames of the development of plastic (between 80˚C and 250˚C). Subsequently, the behavior of the polymer blend powder (PVC) (with its shaping additives) is studied as well the load of well-cured palm kernel husk powder to understand the state of the plastic material obtained with this mixture. Finally, they were tried to understand the behavior of the plastic material resulting from this mixing powder in its total degradation by studying the beha-

vior of the polymer (PVC) on one hand and the shell powder on the other under hand in the same conditions.

The Figure 5(a) & Figure 5(b) below, present the thermogravimetric and differential scanning calorimetry of raw palm kernel shell between 0˚C and 250˚C.

Figure 5 tells us about the thermal behavior on the total degradation of palm kernel shell powder between 0˚C and 250˚C. Literature says that most polymers have their shaping temperature below 250˚C. From the Figure 5(a), it appears that the water starts to evaporate around 70˚C, this could be attributed at free water. After the departure of water, around 189˚C the constituents of raw material begin to degrade. This temperature corresponds to degradation of hemicelluloses polymer. At this temperature, cellulose and lignin are not degraded. At this temperature, several polymers have already exceeded their processing temperature. Thus, cellulose and lignin shell still have all their properties to offer to the plastic (PVC) all the possibilities to have good resistance.

Similarly, Figure 5(b) shows that in its degradation process in the temperature range that surrounds the shaping temperature of the polymers. It can be observed that outside free water at 101˚C the palm kernel shell still retain their structure at more than 80% of their initial mass. Hemicellulose is infinitely small (TG), cellulose begins to absorb heat to enter total degradation (360˚C). This justifies that the shell powder still keeps the maximum of its crystalline and chemical structure. Then a mixture of polymer with palm kernel powder will bring a very good improvement of several properties of the polymer.

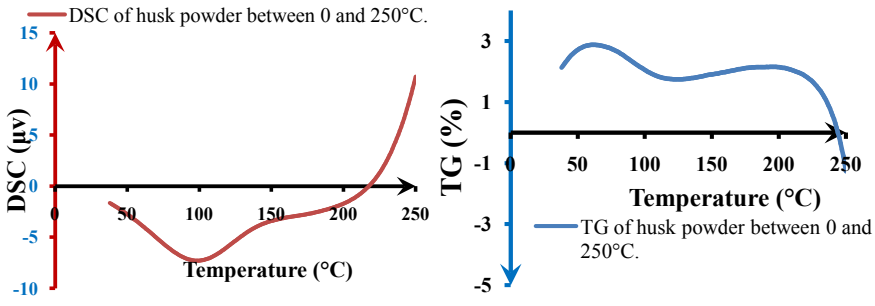
3.7. Results of PVC

Characteristic parameters:

The PVC used for this study comes from DANSUK INDUSTRY4. It was delivered to us in a 25 kg bag in the form of a white powder (KN 500).

The TG and DSC of raw PVC are presented in Figure 6.

Figure 6 shows the mass loss (TG) (in red) and the differential scanning calorimetry (DSC) in blue of the raw PVC, for a temperature velocity of 10˚C/min at atmosphere controlled by oxygen. In this figure, it appears that there are



(a) (b)

Figure 5. Thermogravimetric (a) and differential scanning calorimetry (b) of raw palm kernel shell between 0˚C and 250˚C.

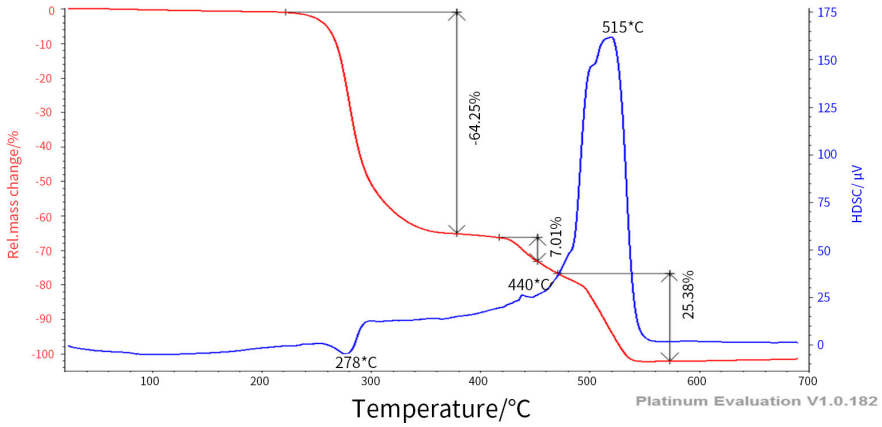


Figure 6. Thermogravimetric and differential scanning calorimetry of raw PVC.

three main phases of degradation of the raw PVC:

1) Dechlorination phase: the degradation starts at 220˚C and leads to about 64.25% of mass loss. This phase corresponds to the degradation of HCl and the formation of polyene structure.

2) Condensation phase: this phase starts at 420˚C and end at 470˚C and leads to about 7.01% of weight loss. In this phase, a part of polyene is degraded and the other part of the polyene molecules rearrange through cyclization reactions and crosslinking by forming aromatic hydrocarbons and ashes.

3) Fragmentation phase: this phase starts at 470˚C and leads to about 25.38% of weight loss. The aromatic compounds formed before are degraded, all the material being degraded, only the ashes remain.

These results are in agreements with the results obtained by others researchers [4] [30].

The DSC shows an endothermic peak of heat at 278˚C. During the degradation of polyene and aromatic compounds an exothermic is observed at 440˚C and 515˚C. It can also be noted that the ignition and the burnout temperatures for the raw PVC are around 220˚C 560˚C respectively.

The DSC of PVC was also done in order to determine the glass transition temperature (Tg). This is an important parameter for polymer characterization because it permits to evaluate the plasticizing effects of substances when it is added on polymers. The curve of the DSC of PVC is presented in **Figure 7** in the range between 0˚C to 250˚C.

As we can observe in **Figure 7**, we an endothermic peak between the ranges of the temperature chosen. From this curve, it appears that the glass transition temperature of pure PVC is 98.56˚C which is in the glass transition temperature range found in literature for this polymer [4] and the melting temperature is 147.84˚C [30].

3.8. Results of the PVC Blending Powder and 12. 54% of the Palm Kernel Shell Powder

After the study of raw palm kernel shell and PVC, we made a composite loaded

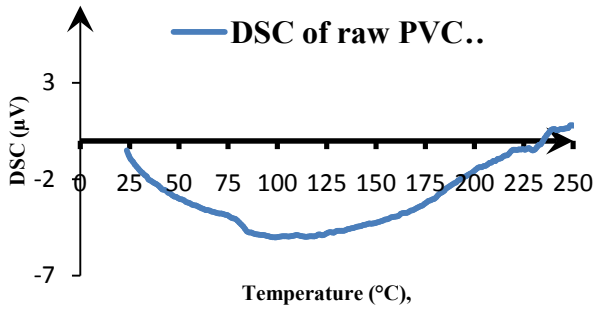


Figure 7. Differential scanning calorimetry of raw PVC at the temperature between 0˚C to 250˚C.

at 12.54% with palm kernel shell. The Figure 8 presents the thermogravimetric and Differential scanning calorimetry of the composite.

The Figure 8 is the TG in red and DSC in blue of the composite (PVC + 12.54% of the palm kernel powder). The TG shows that there are three peaks of the thermal degradation of the composite:

1) Dehydration: the degradation starts at 98˚C and leads to about 1.02% of mass loss. This phase corresponds to the dehydration of the material.

2) The second phase of degradation starts around 235˚C and end around 360˚C, consist of the dechlorination of PVC, the degradation of cellulose and hemicelluloses structures. This phenomena leads to a mass loss of 64.21%. In this phase there is also a rearrangement of the composite and production of the ashes. Compared with the TG of palm kernel shell and PVC at 360˚C, there is a decrease in mass equivalent to 64.21% of the dry mass against 64.25% for PVC and 48.48% for palm kernel shell. This influence is weak because the loading rate of the palm kernel shell powder is low, hence the behavior of the TG tend towards the TG of PVC.

3) The third phase of degradation starts at 360˚C and leads to 20.82% of weight losses. This phase consist of degradation of lignin and residual residue and the production of ashes that shows the total combustion of the composite. We can also note that the ignition temperature is around 98˚C and the burnout temperature is 540˚C for the composite.

The DSC curves (Figure 8) shows an endothermicpeak between 90˚C and 95˚C corresponding to the glass transition phase of the composite. We note from the DSC that the glass transition temperature is 95˚C against 98.65˚C for PVC. After this temperature, the heat evolution flux is progressive up to its crystallization start temperature which is about 391˚C. Similarly, the mixture enters total crystallization where it peaks at 459˚C. The residue is deposited at the bottom of the crucible and absorbs heat near 478˚C where it crystallizes for the second time leaving the ash at 556˚C. Surely, the residues come from the mixture of shell powder and PVC that have been calcined.

By comparing the different DSC, it can be observed that palm kernel powder absorbs more heat than PVC. This phenomenon can be justified by heat absorption

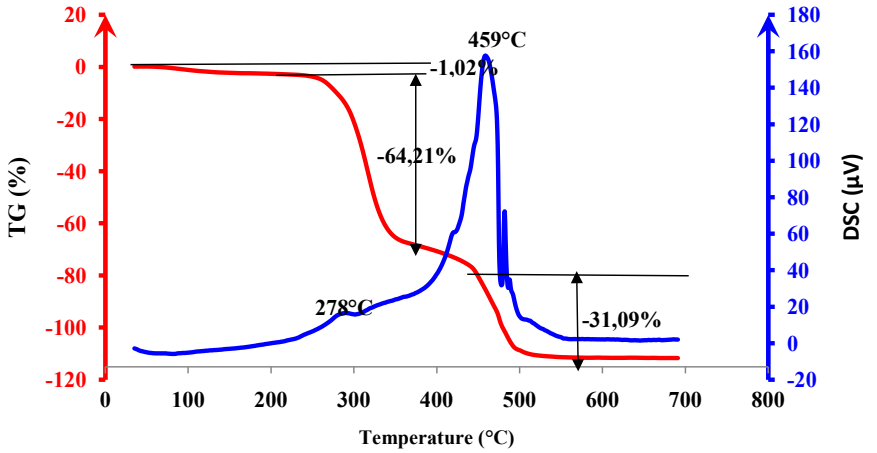


Figure 8. Thermogravimetric and Differential scanning calorimetry of the composite.

of carbon [6] [21] contained in the powder. This parameter allows therefore, taking precautions in the setting of the temperature (170˚C to 205˚C for PVC) of the machine at the time of shaping the PVC mixture loaded with palm kernel shell powder. This heat absorption of the powder influence the parameters such as the glass transition temperature (95˚C), the melting temperature (149˚C), the start temperature of crystallization (409˚C), the peak temperature of crystallization (459˚C) of the elaborated plastic material [2] [24].

One of the important parameters of a material is its degradation as a function of the increase or decrease of the temperature. After the study of the TG and DSC of palm kernel shell, PVC and composite, Hence, the next and important stage, was to see how the material absorbs heat during the weight loss. The result is presented in **Figure 9**.

From the **Figure 9**, it can be note that, the shell powder absorbs heat very quickly with a mass loss around 40% then, conserves this heat until its total degradation. This quickly absorption of heat can be attributed to the cellulose and hemicelluloses content in palm kernel powder. On the other hand, PVC slowly absorbs the heat but quickly loss mass when is hot up to about 75%. Between these two behaviors described, the composite obtained presented an intermediate behavior. The composite obtained absorbs moderately heat and have a mass loss around 60% and enters crystallization zone. Following these findings, one can say that palm kernel powder greatly influences the plastic material especially if it is used at a high percentage. This phenomenon of the conservation of the heat by a material offers an advantage to the palm kernel shell powder today if it is used as load during the fabrication of clothes for winter, for sailors especially for the regions where cold predominates. It can also be used as a coating (paint) for the hot storage of products to name just these examples.

In **Table 2**, we presented the different temperature obtained.

Table 2 shows that the glass transition temperature went from 98.56˚C for PVC to 95˚C for the composite which brings the melting temperature to 147.78˚C for PVC to 142.5˚C for the composite. It is thus noted that there has

Table 2. Impact of palm kernel powder on the phase change temperatures.

	Glass transition temperature (Tg)	Melting temperature (Tm)	Crystallization start temperature (Tec)	Peak crystallization temperature (Tc)	Ashes start temperature (Ta)
PVC	98.56˚C	147.84˚C	455.56˚C	519.5˚C	556˚C
Composite	95˚C	142.5˚C	368˚C	459˚C	558˚C

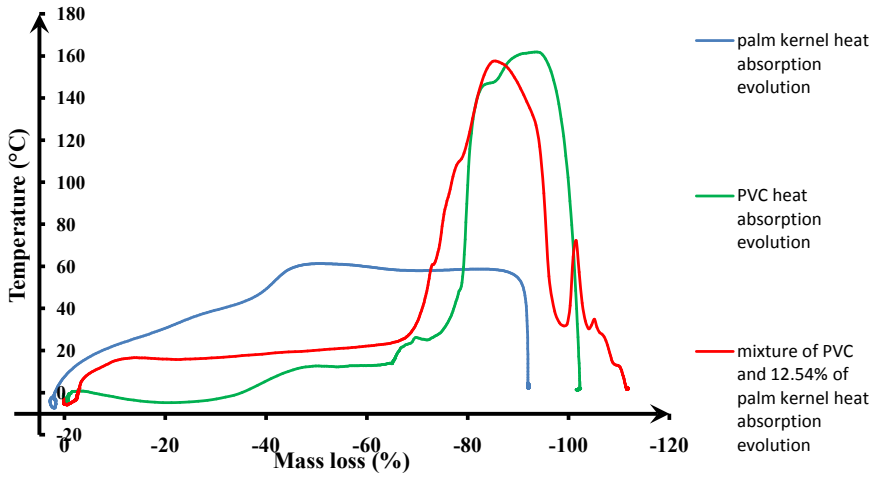


Figure 9. Absorption of heat during the mass loss of different materials.

been a decrease in the glass transition temperature of PVC. It can thus be concluded that the palm kernel shell reinforces the plasticizing effect like all natural plasticizers. Then, we obtained the crystallization start temperature at 455˚C for the PVC against 368˚C for the composite, which gives the crystallization peak temperature of 519˚C for the PVC against 495˚C for the composite. Finally the ash is started to be obtained around 556.56˚C for the PVC against 558˚C for the composite.

4. Conclusion

In this study we transformed palm kernel shell to palm kernel powder. We have studied the possibility of using palm kernel powder as filler for polymers and using PVC as polymer. The results show us that the load of palm kernel shell powder greatly reduces the phase temperatures of the polymers. In the same way, the palm kernel shell load decreases the mass loss of the PVC loaded up to the melting point of the PVC, but in the crystallization phase of PVC, the loss of mass decreases to its degradation. From the thermogravimetric curve of palm kernel shell, we observed three levels of degradation of raw matter with 1.13%, 48.38% and 20.82% of weight loss respectively. These different mass losses have attributed to the dehydration (1.13%), hemicelluloses and cellulose (48.38%) and lignin (20.82%). The composite made by filler PVC with 12.5% of palm kernel shell permits us to conclude that, the behavior of composite is between the behavior of PVC and palm kernel shell powder. The diminution of glass transition

temperature ensures us that we can use the palm kernel shell powder to produce new material with news performances.

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