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SEM-BASED STRUCTURAL AND CHEMICAL ANALYSIS OF PARAFFIN WAX AND BEESWAX FOR PETROLEUM APPLICATIONS

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ABSTRACT

In this study, paraffin wax and beeswax are used in the laboratory to simulate the reservoir rock. The physical and chemical analyses of beeswax and paraffin wax have been conducted using Energy Dispersive X-ray analysis (EDX) coupled with scanning electron microscope (SEM). Experimental results show that in paraffin wax, the composition of carbon varies from 10.33 to 12.99 (as weight basis 34.42 % to 45.14%) and oxygen varies from 0 to 0.13 (as weight basis 0 % to 1.95 %). For beeswax samples, the concentration of carbon varies from 11.14 - 17.62 (as weight basis 42.82 % to 68.17%) and oxygen ranges from 0.40 to 0.67 (as weight basis 5.45 % to 9.10 %). SEM analysis identified that carbon and oxygen are the main components in paraffin wax and beeswax samples. The resulting micrographs indicate the morphology of the beeswax and paraffin wax. The micrographs of paraffin wax show that this type of wax consists of two distinctive shades, one darker with long chains and smaller oblongate shape, and lighter pattern which is background. The micrographs of beeswax samples reveal topographic variations on the wax surface including ridges and valleys.

Keywords: SEM; EDX; paraffin wax; beeswax; physical and chemical properties; material structure.

1. INTRODUCTION

The physical and chemical properties of a rock are very important in any reservoir study. In laboratory, it is very hard to drill a real reservoir rock with a waterjet using the normal available laboratory facilities. There are two ways to do such an experiment in laboratory. A

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very high energy pump can be used to develop a very high pressure to cut the rock sample. A soft sample can be drilled with low pressure. It is easy to accept the second option because it suits the laboratory situation. Then the soft sample can be employed to simulate a real reservoir rock sample.

The use of hard rock drilling or excavation machines is increasing in recent years in the field of drilling, mining and civil engineering. Therefore, it is important to pay our attention to improve the existing technology or look forward an alternative of drilling technique. In existing technology, the questions about the ability of drilling machines to operate and cut effectively in hard rock are well known (Bilgin et al., 2006). These difficulties are limited by the system stiffness and the ability of cutting tools to withstand high forces. Moreover several factors such as cutting forces, force acting on a cutting tool, chipping out, and brittle nature of rock are the governing criteria for drilling in rock formation. The high force acting on formation rock may damage the machine components and exceed the machine's torque and thrust capacities.

Therefore, it is essential to overcome these shortcomings in drilling technique. Waterjet drilling is one of the competitive and reliable techniques in replacing this conventional drilling method. To analyze the effectiveness and sustainability of waterjet as a drilling technique, a series of laboratory tests were conducted and justified the results (Hossain et al., 2007a; Hossain et al., 2007b; Hossain et al., 2007c; Hossain et al., 2007d).

Morgan et al. (2002) studied the mechanical properties of beeswax and measured these properties as a function of temperature. They used a variety of techniques and compared with each other. In the study, the coefficient of friction of beeswax was measured and compared with that of plasticine and Nylon 6–6. They found that the frictional behavior of beeswax departs from Amonton's law and behaves instead as a classic soft, elastic polymer. Amonton's law states that the pressure of a gas is directly proportional to the temperature (Kelvin) at constant volume, V and number of moles, n.

This paper analyzes the paraffin wax and beeswax samples utilizing scanning electron microscope (SEM) coupled with Energy Dispersive X-ray analysis (EDX). It establishes their main components and morphology. This paper studies the different physical and chemical properties of paraffin wax and beeswax that will be used to simulate rock drilling in the field.

2. PARAFFIN WAX

Paraffin wax is a tasteless and odorless white translucent solid. The source of paraffin wax (see Figure 1) is petroleum as indicated in Figure 2 presenting its pathway. It is produced following petroleum refining by dewaxing light lubricating oil stocks. It consists of a mixture of solid aliphatic hydrocarbons of high molecular weight such as $C_{36}H_{74}$. Its molecular formula is C_nH_{2n+2} (Lewis, 2002). Paraffin wax can be defined as a fraction of petroleum dominated by n-alkanes that are solid at ambient temperature (Chouparova and Philp, 1998). It contains above C_{8+}, smaller amounts of isoalkanes, cycloalkanes and aromatics. Paraffin waxes are chemically stable and have a negligible degree of sub cooling during nucleation. There is no phase separation, and the phase change process only results in a small volume change (He et al., 2004). Paraffin waxes are commonly classified in the petroleum industry literature as paraffin, intermediate and microcrystalline type (Jowett, 1984; Speight, 1991).

Figure 1. White paraffin wax sample used in lab for SEM tests.

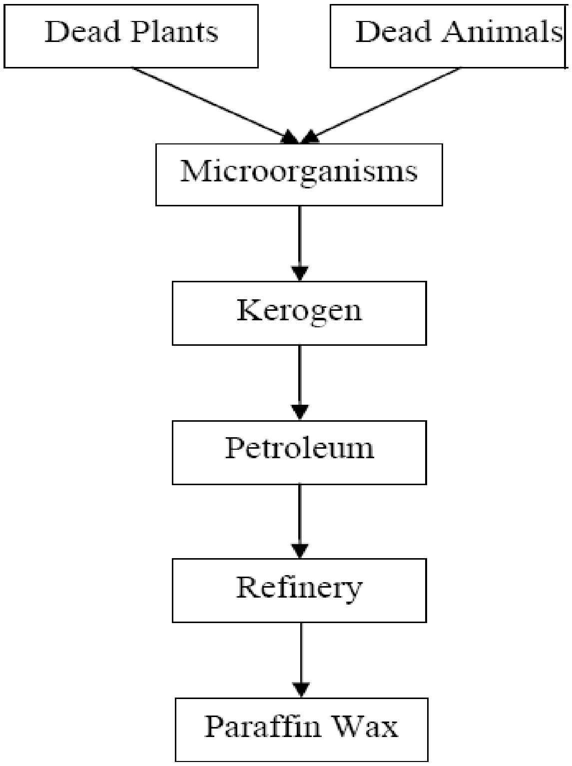

Figure 2. Paraffin wax pathway.

Table 1 shows the paraffin wax properties including the values of wax density, melting point, flash point, and autoignition temperature. The density varies between 0.88 and 0.94 g/cm^3 (Lewis, 2002; Krupa and Luyt, 2001). The melting point ranges from 47 to 65°C

(Lewis, 2002). The flash point equals 390°F or 198°C (Lewis, 2002). The autoignition temperature is reached at 473°F or 245°C (Lewis, 2002). The paraffin wax has a molar mass equal to 785 g/mol and a C/O ratio of 18.8/1 (Krupa and Luyt, 2001). Its common properties are water repellency, smooth texture, low toxicity, and freedom from objectionable odor and color (Speight, 1991). Paraffin waxes contain carcinogens since they are processed using toxic material. The threshold limit value for paraffin wax is 2 mg/m^3 (Lewis, 2002). Paraffin wax is soluble in benzene, ligroin, warm alcohol, chloroform, turpentine, carbon disulfide, and olive oil. It is insoluble in water and acids. Paraffin wax is combustible and has good dielectric properties. Paraffin wax grades are yellow crude scale, white scale, and refined wax. Paraffin waxes are also graded by melting point and color. The higher-melting grades are more expensive. Table 2 shows the classification of paraffin waxes and their properties.

Paraffin wax is used in the manufacture of candles, paper coating, protective sealant for food products and beverages, glass-cleaning preparations, hot-melt carpet backing, biodegradable mulch (hot melt-coated paper), impregnating matches, lubricants, crayons, surgery, stoppers for acid bottles, electrical insulation, floor polishes, cosmetics, photography, antifrothing agent in sugar refining, packing tobacco products, protecting rubber products from sun-cracking, and chewing-gum base. Waxy substances have been transformed and used by our ancestors as early as the neolithic period for a large range of activities (Regert et al., 2005). They used as waterproofing substances, illuminant, sealing agent but also for adhesive making and many other technical, medicinal or symbolic purposes. Additives are often mixed with these waxy materials in order to improve their properties. Resins are used to harden and colour the material. Fatty materials increase malleability and softness of waxes. Pigments and dyes colour the material and starch is used as an extender.

Table 1. Wax properties

Wax type	Density (g/cm^3)	Melting point (°C)	Flash point °F (°C)	Autoignition temp °F (°C)
Paraffin wax	0.88-0.94[1,2]	47-65[1]	390 (198)[1]	473 (245)[1]
Beeswax	0.95[1]	62-65[1]	-	-

Sources: 1 Lewis (2002) and 2 Krupa and Luyt (2001).

Table 2. Classification of paraffin waxes

Properties	Paraffin wax	Amorphous wax
N-alkanes dominant range	$C_{18} – C_{30}$	$> C_{18} – C_{30}$
Amount of other HC (iso-, cycloalkanes, etc)	Lower	Higher
Melting point range	40 – 60°C	> 60 – 90°C
Adhesion	Lower	Higher
Source fractions	Light distillate	Heavy distillate, residual oil, pipeline and tank wax deposits

Source: Chouparova and Philp (1998).

3. BEESWAX

Beeswax is a type of wax from the honeycomb of the honeybees (see Figures 3 and 4). It is yellow, brown, or white bleached solid. The normal color in the wax gives shades of yellow, orange and red through to brown.

Figure 3. Yellow beeswax sample used in lab for SEM tests.

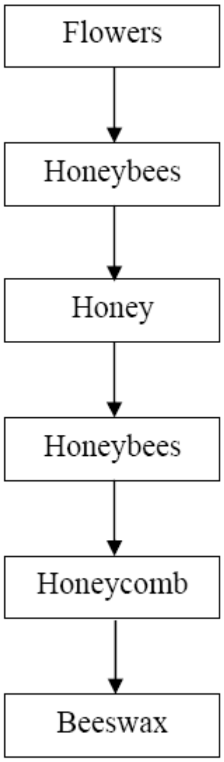

Figure 4. Beeswax pathway.

The colour of beeswax changes with age, for example virgin wax is white but darkens rapidly as it ages, often becoming almost black. It has a faint honey odor. It consists largely of myricyl palmitate, cerotic acid and esters, and some high-carbon paraffins. Beeswax is lipid by nature. It has saturated hydrocarbons, acids or hydroxy-acids, alcohols, pigments, mostly from pollen and propolis, as well as minute traces of brood etc. (Leclercq, 2006). Beeswax has a very stable chemical make-up, alcohols, pigments, mostly from pollen and propolis, as well as minute traces of brood etc. (Leclercq, 2006). Beeswax was the earliest waxy material exploited by men (Regert et al., 2005). However, many other natural substances have been used thereafter. Chinese insect wax, shellac wax, spermaceti and wool wax, from animal origin; carnauba, candelilla or Japan waxes, secreted by various plants, and fossil materials (Regert et al., 2005). Beeswax is the natural wax made by honey bees in the hive and is also known as Cera alba and Cera flava (Columbus Foods, 2002).

Table 1 shows the beeswax properties including the values of wax density and melting point. Its density equals 0.95 g/cm^3 (Lewis, 2002; Leclercq, 2006). Its melting point ranges from 62 to 65°C (Lewis, 2002; Columbus Foods, 2002). It is completely insoluble in water due to its resistance to hydrolysis and natural oxidization. However, it is soluble in alcohol, chloroform, ether, and oils. It is combustible. The properties of beeswax remain unspoilt by time. Apart from the larvae of the wax moth, no animal has the digestive acids and juices to break it down (Leclercq, 2006). It is solid in appearance in normal temperatures. It becomes brittle when the temperature drops below 18°C however it quickly becomes soft and pliable at around 35° to 40° C. Beeswax grades are technical, crude, refined, NF (National Formulary grade of chemical), FCC (Food Chemical Codex), and white USP (United States Pharmacopeia). It is used in the manufacture of furniture and floor waxes, shoe polishes, leather dressings, anatomical specimens, artificial fruit, textile sizes and finishes, church candles, cosmetic creams, lipsticks, and adhesive compositions. A wide variety of cosmetics use beeswax as an emulsifier, emollient, and moisturizer. It is often used in skincare products as a thickening agent. After processing, beeswax remains a biologically active product retaining anti-bacterial properties. It also contains vitamin A, which is essential for human cell development. Throughout time, people have used it as an antiseptic and for healing injuries. Beeswax has many other industrial uses too.

4. SCANNING ELECTRON MICROSCOPY

Scanning electron microscopy is the best known and most widely-used of the surface analytical techniques. SEM, accompanied by X-ray analysis, is considered a relatively rapid, inexpensive, and basically non-destructive approach to surface analysis (Thipse et al. 2002). It is often used to survey surface analytical problems before proceeding to techniques that are more surface-sensitive and more specialized. SEM with or without energy or wavelength dispersive X-ray detectors for chemical analysis (also known as microprobe or electron probe micro analysis) has been and still is routinely used in the study of rocks and minerals. In order to use the SEM and the microprobe facilities the samples have to be coated with a thin conducting layer of gold in order to prevent charging of the sample (Deydier et al., 2005). Figure 5 shows the Scanning Electron Microscope (SEM) used in our wax material studies. It is important here to note that this SEM cannot detect the sample elements that are less than 2% by weight.

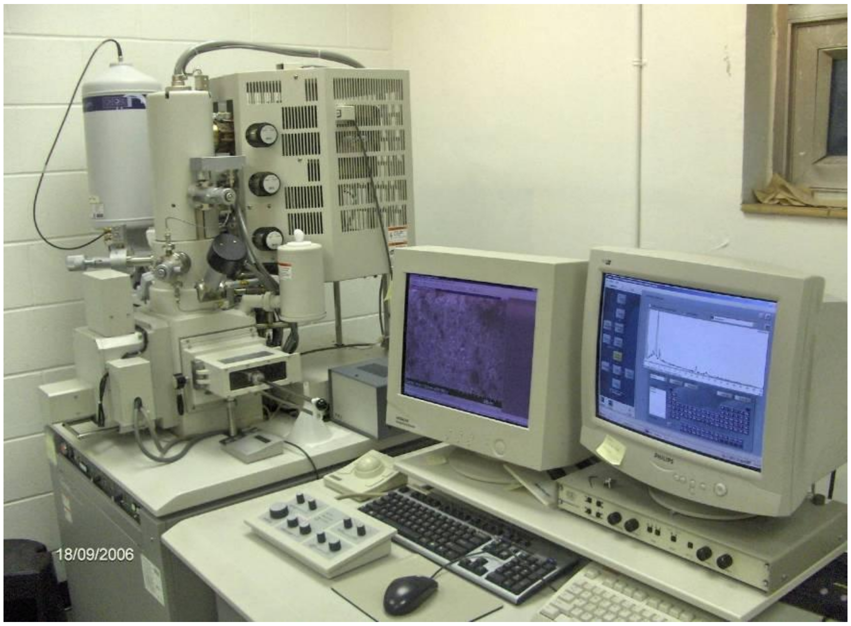

Figure 5. Scanning Electron Microscope (side view)

5. PREPARATION OF SAMPLES

In order to view non-conductive samples, SEM's require that the samples be electrically conductive (Hu et al., 2004). To perform our analysis, bees wax and paraffin wax samples were coated with a thin layer of conductive material gold (Au). To do this a small device called a sputter coater was used. The sputter coater uses argon gas and a small electric field. The bees wax and paraffin wax samples were first fixed on double sided adhesive carbon tapes attached to one end of SEM stubs and then the samples were attached with that carbon tapes. Then the sample is placed in a small chamber which is at vacuum. Argon (Ar) is then introduced and an electric field is used to cause an electron to be removed from the argon atoms to make the atoms ions with a positive charge. The Argon ions are then attracted to a negatively charged piece of gold foil. The Argon ions act like sand in a sandblaster, knocking gold atoms from the surface of the foil. These gold atoms now settle onto the surface of the sample, producing a gold coating. The thickness of coating was 30.00 nm, density 19.32 g/cm^3. The coated samples were investigated using a SEM (Hitachi S-4700, Japan) equipped with an EDS system (INCA, UK) and an accelerating voltage of 20 keV for spectrum analysis. The experimental condition of SEM-EDX is given in Table 3 and the sample coating is shown in Figures 6 and 7.

Table 3. Experimental condition of SEM-EDS for paraffin wax and beeswax

Particulars	Paraffin wax	Beeswax
Type	Default	Default
Live time	100 sec	100 sec
Real time	104.4 -104.59 sec	104.68 - 105 sec
Acquisition geometry (°)	Tilt = 0.0	Tilt = 0.0
	Azimuth = 0.0	Azimuth = 0.0
Spectrum processing	No peak omitted	No peak omitted
Number of iteration	3	2-3
Processing option	All elements analyzed	All elements analyzed
Detector	Silicon	Silicon

Figure 6. A sputter coater coats the sample with gold-palladium atoms.

Figure 7. Coated paraffin wax and beeswax samples for SEM tests.

6. RESULTS AND DISCUSSIONS

For the physical and chemical characterization of solid materials, SEM is one of the best and most widely used techniques (Vassilev and Vassileva, 2005). SEM, using a focused electron beam to scan the surface of a sample, generates a variety of signals. The three most common modes of operation in SEM analysis are BackScattered Electron imaging (BSE), Secondary Electron Imaging (SEI), and EDS (Postek et al., 1980). In this study, EDS (Energy-dispersive X-ray Spectrometry) coupled with SEM were used to characterize paraffin wax and beeswax samples. The elemental analysis was performed in a spot mode in which the beam is localized on a single area manually chosen within the field of view. The EDS detector was capable of detecting elements with atomic number equal to or greater than six. The intensity of the peaks in the EDS is not a quantitative measure of elemental concentration, although relative amounts can be inferred from relative peak heights (Kutchko and Kim, 2006).

In order to examine the internal structure and composition of paraffin wax and beeswax, each sample was characterized by randomly selecting 3 fields of view and examining the samples. The SEM data (see Tables 4 and 5) clearly indicate that the main composition of paraffin wax and beeswax are carbon and oxygen that are shown in the Figures 8 to 12.

Table 4. SEM experimental test results for paraffin wax

Particulars	Spectrum 1			Spectrum 2		
	C	O	Total	C	O	Total
App. concentration	12.99	0.13	--	10.53	0.00	--
Intensity corrn.	0.2877	0.0667	--	0.3058	0.0635	--
Weight (%)	45.14	1.95	47.09	34.42	0.00	34.42
Weight (%), sigma	0.96	0.68	--	0.83	0.00	--
Atomic (%)	96.86	3.14	--	100.00	0.00	--

Table 5. SEM experimental test results for beeswax

Particulars	Spectrum 1			Spectrum 2			Spectrum 3		
	C	O	Total	C	O	Total	C	O	Total
App. concentration	17.62	0.67	--	12.30	0.44	--	11.14	0.40	--
Intensity corn.	0.2584	0.0736	--	0.2599	0.0732	--	0.2601	0.0731	--
Weight (%)	68.17	9.10	77.28	47.34	6.07	53.40	42.82	5.45	48.27
Weight (%), sigma	1.23	0.97	--	1.04	0.86	--	0.98	0.80	--
Atomic (%)	90.89	9.11	--	91.22	8.78	--	91.28	8.72	--

The composition of carbon varies from 10.33 - 12.99 (as weight basis 34.42 % to 45.14%) and oxygen is varies from 0 to 0.13 (as weight basis 0 % to 1.95 %). For beeswax samples, the concentration of carbon varies from 11.14 - 17.62 (as weight basis 42.82 % to 68.17%) and oxygen varies from 0.40 to 0.67 (as weight basis 5.45 % to 9.10 %).

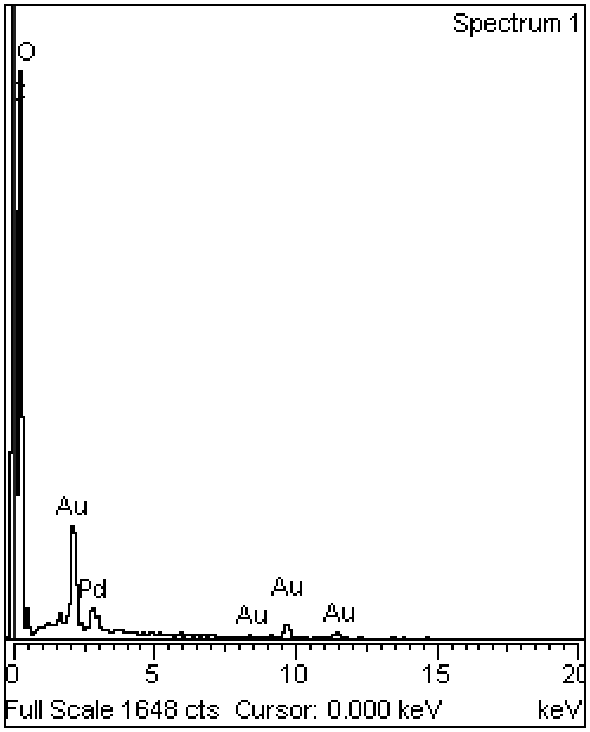

Figure 8. Spectrum analysis for paraffin wax for Run 1.

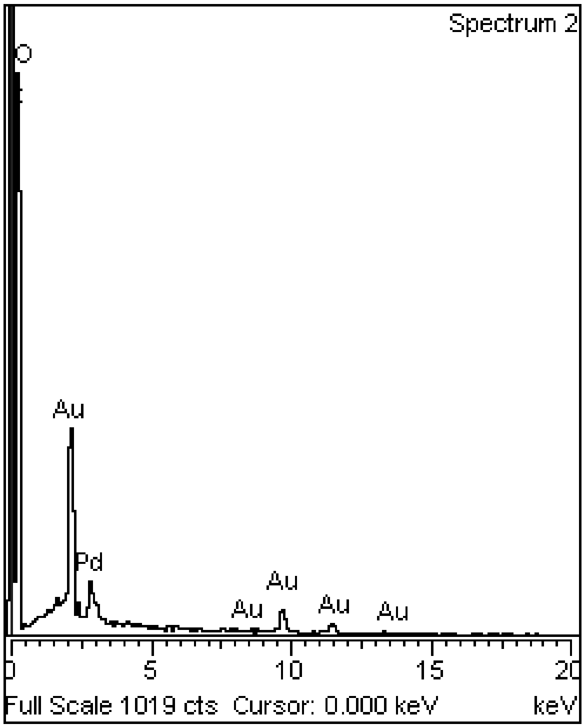

Figure 9. Spectrum analysis for paraffin wax for Run 2.

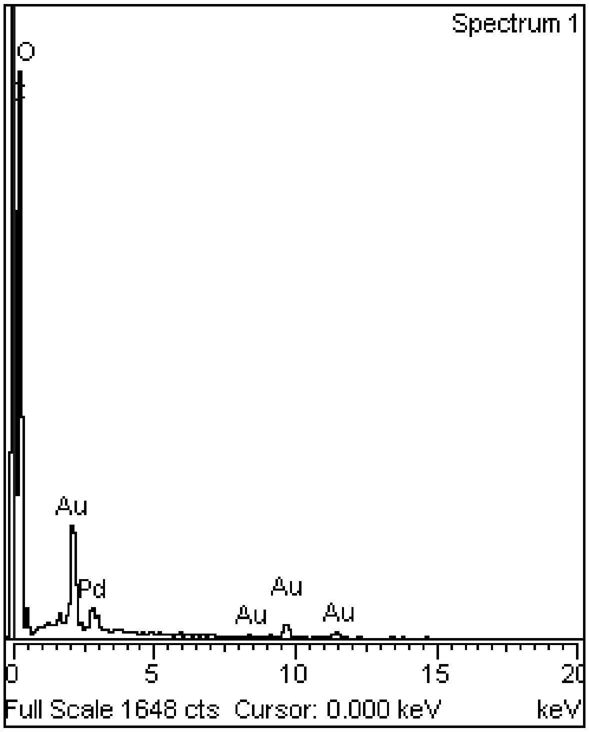

Figure 10. Spectrum for beeswax for Run 1.

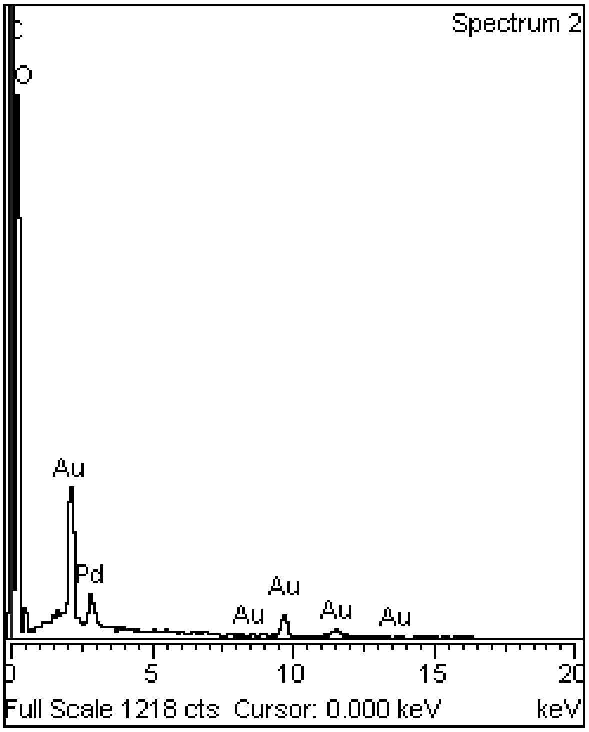

Figure 11. Spectrum for beeswax for Run 2.

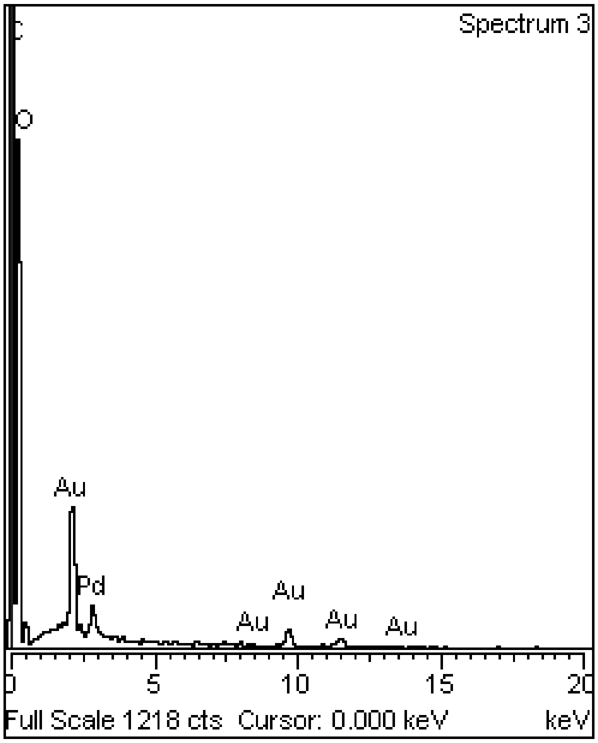

Figure 12. Spectrum for beeswax for Run 3.

EDS microanalysis of all spectrums in the paraffin wax and beeswax samples confirm the presence of carbon and oxygen as illustrated in Figures 8 to 12. The other peaks appearing in those figures might be due to the sample coating process, which indicates the presence of gold, palladium, or both.

7. STRUCTURAL ANALYSIS OF PARAFFIN WAX AND BEESWAX

Sample handling, coating and preparation for SEM cause sample alteration, which modifies its composition and morphology structure. Therefore the quality of the micrographs obtained is affected as well. Figures 13 to 15 show the SEM micrographs for paraffin wax following three magnifications of 250, 1050, and 2000 respectively. Some micrographs are affected by charging that alters the brightness and contrast levels. For example, the bright spots in Figure 13 exhibit the charging effect due to the presence of electrons that did not penetrate the wax. Figures 16 to 18 illustrate the SEM micrographs for beeswax at three magnifications of 250, 1100, and 2000 respectively. In these figures, the bright spots are more dominant than in the case of paraffin wax, which implies that beeswax is more resistant to electron storming by SEM than paraffin wax. This indicates that beeswax has a lower electric conductivity than paraffin wax.

Regarding the wax morphology, Figures 13 to 15 expose the lamellar structure of paraffin wax, which means that the corresponding sample consists of a blend of polymers.

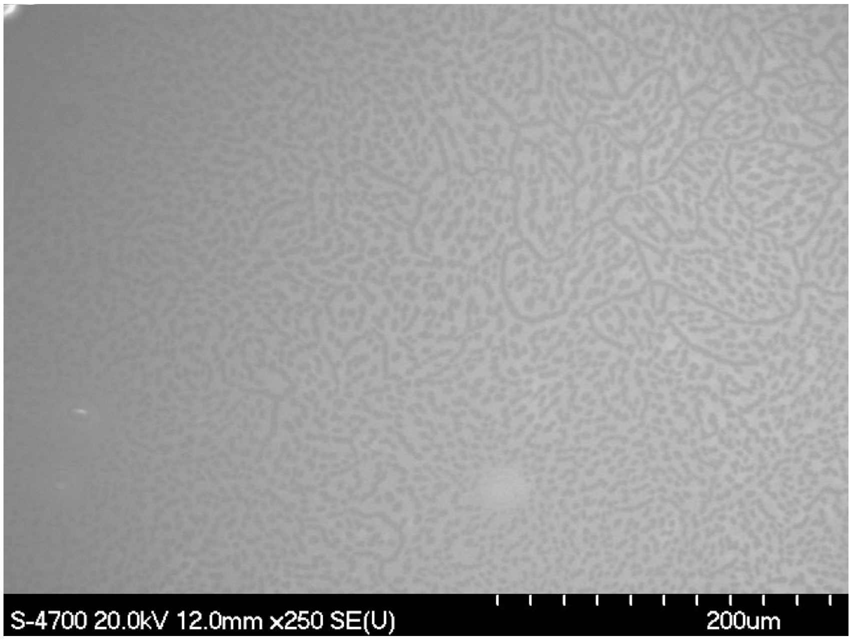

Figure 13. Micrograph of paraffin wax sample, showing two distinctive shades, one darker with long chains and smaller oblongate shape, and lighter pattern which is background Condition: Vacc=20kV, Mag=x250, WD=12mm.

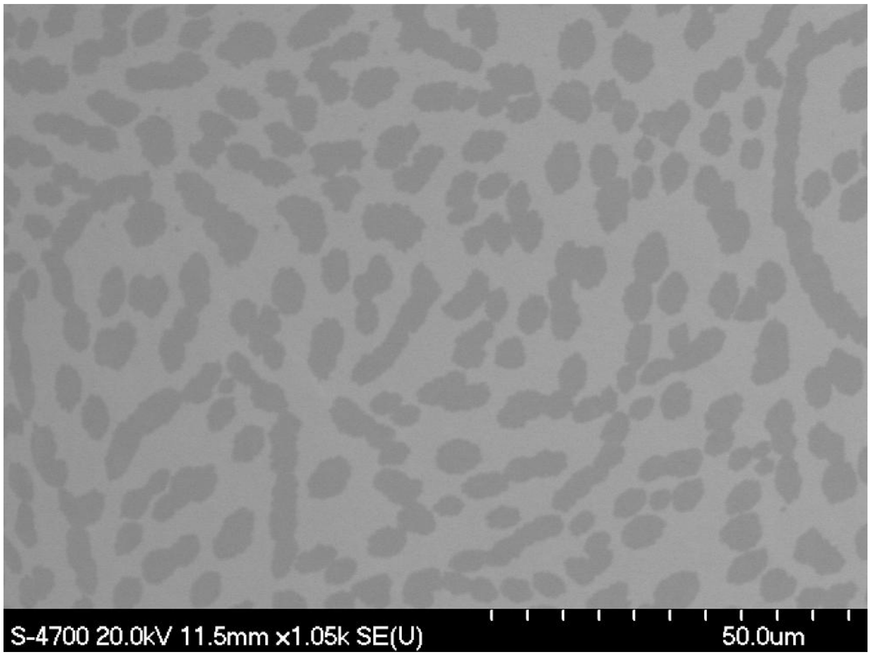

Figure 14. Micrograph of paraffin wax sample in details illustrating the long and short chains, and as well as individual oblongated patterns that are more visible. The background is shown in lighter color. Condition: Vacc=20kV, Mag=x1.05k, WD=11.5mm.

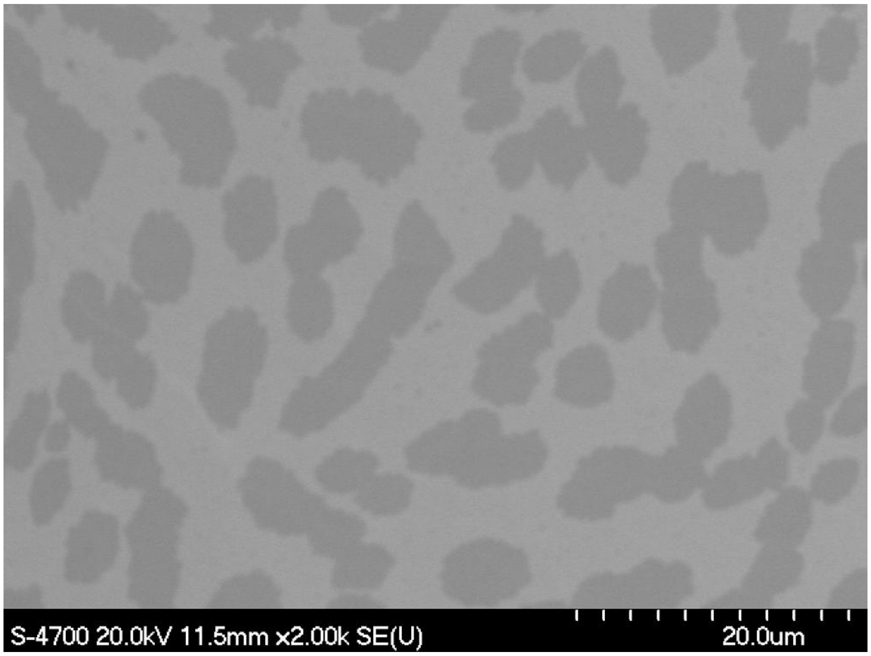

Figure 15. Paraffin wax sample with 2000 magnification indicating that long chains consist of oblongated patterns joining together. Condition: Vacc=20kV, Mag=x2.00k, WD=11.5mm

A miscible blend of amorphous and crystalline polymers means a single phase in the melt and a tidy crystalline phase with a mixed amorphous region in the solid. Because of chain folding during crystallization, the crystal lamellae are formed. Their drastic growth typically leads to the formation of spherulites. Spherulites are ball-shaped spherical masses of radiating crystal fibres. When the miscible blend is submitted to crystallization, the non-crystalline impurity is excluded from the crystalline area.

The paraffin wax is a solid crystalline mixture of solid hydrocarbons of high molecular weight ranging from C_{20} to C_{30} and higher, e.g., $C_{36}H_{74}$. It is derived from the portion of crude petroleum commonly designated paraffin distillate, from shale distillate, or from hydrocarbon synthesis by low-temperature solidification and expression or by solvent extraction. It is distinguished by its solid state at ambient temperatures and relatively slight deformation even under considerable pressures. Paraffin-wax crystals are long and narrow and form in plates. In the fully refined grades they are dry, hard, and glossy. The paraffin wax is characterized by its homogeneous constitution and distribution due to its refining process. Then the separation between the polymers is complete in the paraffin wax samples as indicated in Figures 13 to 15.

The beeswax is secreted by the honeybees in a liquid state at the ambient temperature. Then, it crystallizes at the same ambient temperature. It consists of various components and is characterized by long hydrocarbon chains. It is made largely of a blend of myricyl palmitate, cerotic acid and esters, and some high-carbon paraffins. This reveals the heterogeneous constitution and distribution of beeswax, due to its polymers diversity. According to Figure 16, the beeswax consists of superposed plates. At higher magnifications, the beeswax SEM micrographs (see Figures 17 and 18) display pasty, colloidal, and cloudy structure due to the amorphous and heterogeneous nature of polymers composing the beeswax.

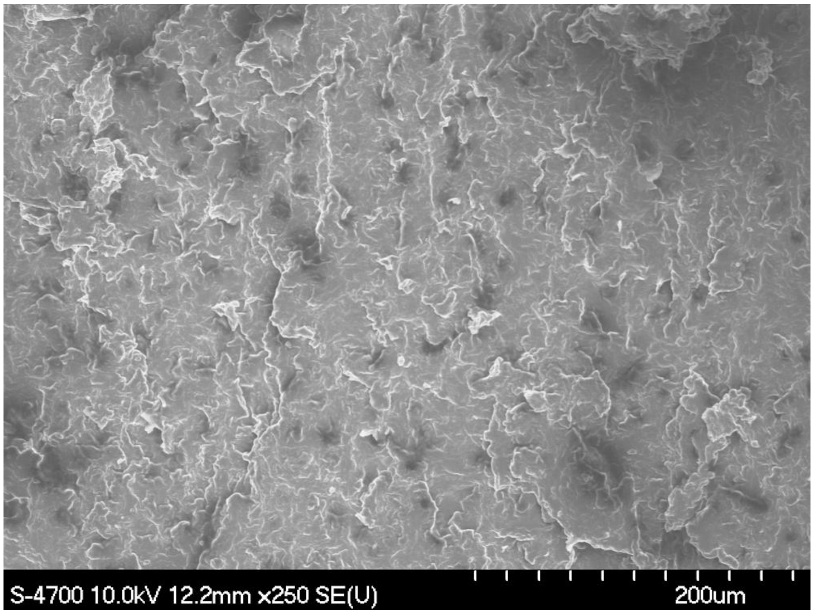

Figure 16. Micrograph of beeswax sample showing topographic variations on the surface. Condition: Vacc=10kV, Mag=x250, WD=12.2mm.

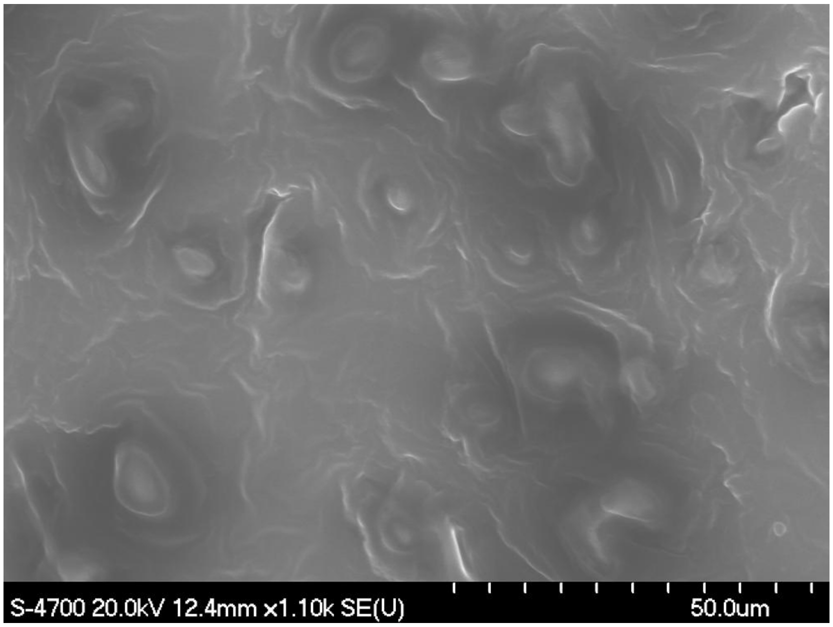

Figure 17. Beeswax sample with 1100 magnification. Closer observation of the beeswax surface with more details showing irregular ridges and valleys. Condition: Vacc=20kV, Mag=x1.10k, WD=12.4mm.

Figures 16 to 18 prove that the multiple polymers in the beeswax did not separate. So these polymers are still solidly interconnected, which explains the beeswax toughness and resistance to electric conductivity. The beeswax is natural. It is younger and fresher than the paraffin wax since the former has a shorter pathway period than the latter (see Figures 2 and

4). The beeswax did not undergo refining stages whereas the paraffin wax was extracted from crude petroleum after refining and purification. This is explained by Figures 13 to 18.

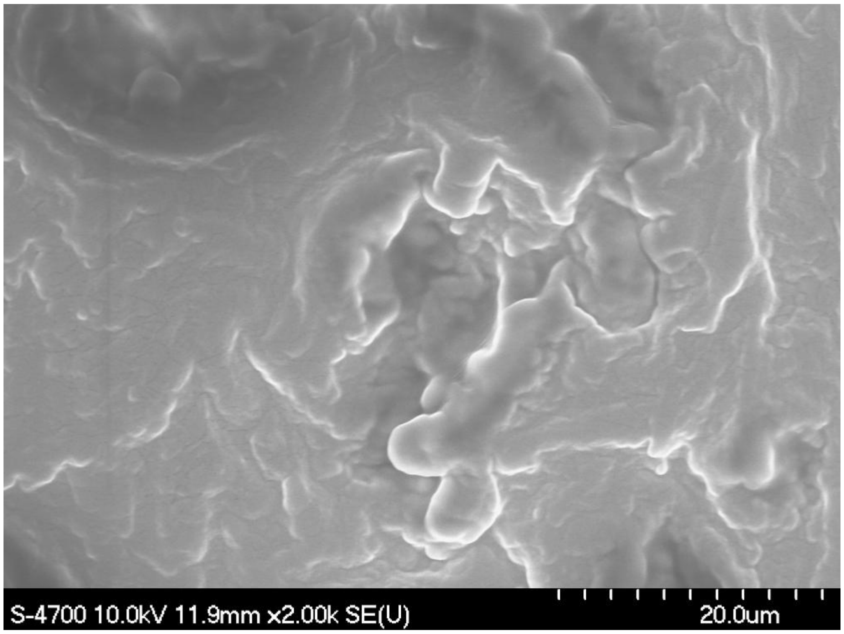

Figure 18. Beeswax sample with 2000 magnification. Detail of the beeswax surface with well visible ridges and valleys, indicating that the sample is not very hard, based on visual observation. Condition: Vacc=10kV, Mag=x2.00k, WD=11.9mm.

CONCLUSIONS

This paper dealing with paraffin wax and beeswax determines their compositions and describes their structure based on spectra and micrographs obtained employing scanning electron microscope (SEM) coupled with Energy Dispersive X-ray analysis (EDX). The various spectra obtained show main presence of carbon and oxygen in both wax types, in addition to contaminants such as gold and palladium due to sample coating and preparation for SEM tests. The micrographs of paraffin wax samples exhibit a perfect distinction between the polymers forming this wax type since it underwent refining and purification which eliminated many wax inherent components and structures. The micrographs of beeswax samples display complex structure due to the beeswax natural state. Beeswax is younger and tougher than the paraffin wax. The former did not suffer refining or purification whereas the latter was submitted to those processes, which eroded its intrinsic components, bonds, and qualities.

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