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# Synthesis, Characterization and Permofarmance Activity of Metal Soap of Calcium, Cobalt, Iron and Copper from Palm Kernel Oil as a Drier in Paint

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**Abstract:** The behavior of Palm Kernel Oil was saponified by potassium hydroxide to form soluble soap which was later reacted with solutions of copper, cobalt, Iron and calcium to produce the metal soaps which were used as drier in coating mixture (Paint). The performance of these metal soaps has been studied. The effect of environmental and corrosive factors on paints was also studied. From the study, the formulated paint samples using metal soap had low specific gravity (1.45-1.47). This is an indication that more metal soap can be incorporated into the paint. Full hardness of paint samples was within the range (0.38-0.41mm) which implies that the samples will have high tolerance to anti-corrosive coating. The paints showed strong adhesion properties that are capable to withstand abrasive and corrosive agents. Also when exposed to surfaces like paper and glass, the exhibited strong adaptability without rust. Considering the drying time of coating mixture (paints) containing metal soaps showed increased oxidizing activity of drier and shortened drying time. The progress of the formulated metal soap paint reaction on storage was monitored and confirmed using physical methods. The high performance of the paints prepared from metal soap is good evidence to justify their utilization as driers in the surface coat industry.

**Keywords:** Palm Kernel Oil, Metal Soap, Saponification, Driers, Synthesis, Metal Soap Paint

## 1. Introduction

Palm kernel oil is an edible plant derived from the kernel of oil palm Elaeisguineensis. It is a member of the family Arecaceae (Palm family). Palm kernel oil (triglyceride) like other nature fats and oil is a chemical compound of one molecule of glycerol, bound to three molecules of fatty acids. It is a semi solid at room temperature is commonly used in commercial cooling because of its relatively low cost and can be stored longer than other vegetable oils.

Presently, petrochemicals are gradually being replaced by oleo chemical feed stocks in many industrial and domestic applications and this has resulted in an increase in demand for bio-based products and as such recognizing and increasing the benefits of using renewable materials. Palm kernel oils are important sources of nutritional oils, industrial and pharmaceutical importance [1], and current emphasis on sustainable development has made it imperative to search for industrial raw materials from renewable sources. Palm kernel oils are at the centre of this search as many useful products and industrial materials have been produced from them. One of such materials is metal carboxylates also referred to as metal soaps.

Metallic soaps have been described as alkaline-earth or heavy-metal long-chain carboxylates [2], which are insoluble in water, but soluble in non-aqueous solvents. Depending on the type of metal salt used, they have been found to form emulsion and slurry respectively. They have the general formula $(RCO_2)_2M$, where M is a metal such as Zn, Cd, Pb, Ba, Ca, Co, Cu, Al, Fe, etc and R is a linear or branched alkyl group. Metal soaps are manufactured by using one of the following processes: double decomposition, direct reaction of carboxylic acid with metal oxides, hydroxides and carbonates and direct reaction of metals with molten fatty acid [3]. These reactions are usually carried out in an aqueous medium for alkaline and alkaline-earth soaps or in an organic medium, such as alcohol or benzene, for metallic soaps [4], [5]. They are used as additive in so many industrial applications. Metal carboxylates have played an important role in the development of poly vinyl chloride (PVC) an important commercial polymer [6]. Other important applications are found in the manufacture of greases, paints or inks, plastics, cosmetics, textiles, pharmaceuticals, etc, in which they are employed as lubricants, driers, catalysts, wetting agents, thickening agents, stabilizers, water- proofing agents, fungicides and pesticides [7], [8]. Many literature sources have revealed excellent report on the characterization and preparation of metal soap [9], their behavior and properties have been studied extensively [10].

Therefore, the present work has added to the available driers beforehand and consequently, they can find uses in paint, varnish, printing ink and linoleum industries. The studies on the nature of these soaps are of great importance for their use in industries and for explaining their characteristics under different conditions. The physicochemical properties of different metallic soaps have been investigated by several researchers. Many of the reports on the characterization and properties of metal soaps have been carried out on soaps prepared using pure fatty acids with little attention on their use as driers, inspite of their abundance, stability and low cost, as starting materials for the preparation of metal soaps. In order to elucidate the drying agents in surface coatings and optimize paint formulations for best cost performance, this work aim to demonstrate the performance of surface coatings (paints) prepared by the addition of metal soap as drier.

## 2. Materials and Methods

Potassium hydroxide, Ca $(NO_3)_2.2H_2O$, Co $(NO_3)_2$, $CuSO_4.6H_2O$, and $FeSO_4$, petroleum spirit, ethanol, sulphuric acid and hydrochloric acid were obtained from BDH Chemicals, England as analytical grade chemicals and used as received.

### *2.1. Chemical Analysis of Palm Kernel Oil*

Palm kernel seeds (50g) were placed in a stoppered container with 1 L n-hexane and allowed to stand at room temperature for a period of 24 h with frequent agitation. The mixture was strained, the damp solid material pressed, and returned into the container. The process was repeated for 7 days to ensure complete extraction. The oil was concentrated by simple distillation which gave 62 and 54% yield for TP and HC oils, respectively. The extracted oil was analyzed for its iodine, saponification, and acid values using the methods described by the Association of Official Analytical Chemists [11].

### *2.2. Fatty Acid Composition of the Oil*

Fatty acid methyl esters of the oils were prepared by saponification according to methods described by [12]. 50 mg of the fat contents was mixed for 5 min at 95°C with 3.4 ml of the 0.5 M KOH in dry methanol. The mixture was neutralized by using 0.7 M HCl. 3 ml of the 14% $BF_3$ in methanol was added. The mixture was heated for 5 min at 90°C to achieve complete methylation process. The fatty acid methyl esters were thrice extracted from the mixture with redistilled n-hexane. The content was concentrated to 1 ml for gas chromatography analysis, 1 µl of this was injected into the injection port of GC which was oven programmed with Model HP 6890 powered with HP Chemstation Rev. A 09.01 [1206] software equipped with flame ionization detector and a capillary column HP INNOWax (30 m × 0.25 mm × 0.25 µm) to obtain individual peaks of fatty acid methyl esters. The inlet and detector temperatures were set at 250 and 320°C, respectively. Nitrogen gas was used and the split ratio was 20:1 with hydrogen pressure at 22 psi. The fatty acid methyl esters peaks were identified by retention times in comparison with calibration curve of the standards analyzed under the same conditions.

### *2.3. Preparation of Metal Soaps of the Oils*

Metal soaps of copper, cobalt, iron and calcium were prepared by methods described by [13]. 50g of palm kernel oil (PKO) was heated to 90-95°C in a 50ml beaker and 125cm$^3$ of 10ml of potassium hydroxide (KOH) emulsified into this thick layer with frequent stirring for one hour. After this period, the soap was then washed with cold distilled water to remove an excess alkali. 0.05M solution of the soap was prepared in hot water and added with stirring to an aqueous solution of the metal salt (0.15Ml). The metallic soap which precipitated out was filtered, washed with distilled water and dried in an air ventilated oven at 40°C for 24 hours. The colours of the metallic soaps were noted. The amount of driers added represents 0.50 wt. % calcium (ca), 0.50 wt. % copper (cu), 0.50 wt. % iron (Fe) and 0.05 wt. % cobalt (Co). These amounts of metals represent those normally used in the surface coatings industry [13]. The amount of driers giving the above percentage of metals in the paint-drier mixtures was calculated as follows:

Amount of drier required

$$= \frac{wt. of\ resin\ in\ paint\ x\ \%\ of\ metal\ required}{\%\ of\ metal\ in\ driver}$$

### *2.4. Property Determination on Prepared Metal Soap Paint Samples*

The properties of the formulated metal soap paint were determined by methods described by [14]. The American standard testing methods (ASTM) was used to evaluate the prepared alkyd paint samples for viscosity (ASTM D 1200-10), specific gravity (ASTM D 1475-13), adherence to surface (ASTM D 6677-07, 2012), settling and skinning tendencies (ASTM D 869-85). The settling tendencies of the samples were rated in accordance to the degree of settling on a scale that range from 10 to 0 (Table 4). Intermediate conditions were given appropriate odd numbers. The Nigerian Industrial Standard (NIS) procedure was used to study the mildew formation resistance (NIS 278:1990), media resistance and chalking tendency (NIS 268:1989) of the prepared metal soap paint samples. Mild steel panels were used as the coating surfaces for the casting of paint-drier mixtures. The panels were prepared according to ASTM D 609-00 procedure, and were taped to 0.40 mm thickness for the casting of the paint-drier mixtures. The paint-drier wet film thickness was assumed the same as the thickness of the tape layer used. The paint-drier mixtures were cast immediately after their preparations on the taped-off portions of the mild steel panels; the wet paint-drier films were leveled off using a short glass rod. The coated plates were placed on a level surface and were allowed to dry at room temperature. Subsequently, the drying properties (surface i.e. dust-free) dry, tack free dry, full hardness and dry for recoat) were studied by finger touch and observation. Adherence to surface: Each of the paint mixture was drawn on the surface of a glass plate to observe color changes on aging. Mildew formation: The paint was observed to see whether there was growth of fungus on the paint film after some days. Skinning, Settling and Gloss: The paint samples were drawn a glass plate for one week with close observation to see if there was any skin formation on the surface of the coat. They were also examined for any sign of deposition of sediments at the bottom for the gloss test, the nature of the finish paint were also determined. Resistance to Water and Heat: The paint film were exposed to rain for two days to observe the resistance of films to wet conditions. Also, to determine their resistance to heat, the samples were coated on a paper and dried in an oven and examined for changes in physical structures.

### *2.5. Color Matching Test*

This was done with the aid of a computer by comparing the color of the resulting paint mixture to identify the colors of each sample. The prime paint without metallic soap (sample 1) was matched with the finish paint (standard or target) on a reflector which was coupled to a computer.

## 3. Result and Discussion

The fatty acids constituents of Palm kernel oil are presented in Table 1. The saponifiable matter of the oil contains different fatty acids. Lauric acid acid had the highest percentage (46%) followed by myristic and oleic acid (15%, 15%) and palmitic acid (8%). Moderate amount of stearic acid (2%), linoleic acid (3%) capric acid (2%) and caprylic acid (1%) were found. Lauric acid is important in the manufacture of soap, the heavy content of lauric acid gives palm kernel oil its sharp melting point nearing hardness at room temperature combined with low melting point [15]. Since Palm kernel oil is packed with lauric and myristic acid, therefore it is suitable for the manufacture of soap.

***Table 1.*** *Fatty acid composition of Palm kernel oil.*

| Name | Retention time | Shorthand | Retention percentage |
|---|---|---|---|
| Capric acid | 10:328 | $C_{10}$:0 | 2% |
| Caprylic acid | 8:219 | $C_8$: 0 | 1% |
| Lauric acid | 13:418 | $C_{12}$:0 | 46% |
| Linoleic acid | 19:516 | $C_{18}$:2 | 3% |
| Myristic acid | 15:014 | $C_{14}$:0 | 15% |
| Oleic acid | 18:827 | $C_{18}$:1 | 15% |
| Palmitic acid | 16:213 | $C_{16}$:0 | 8% |
| Stearic acid | 18:080 | $C_{18}$:0 | 2% |

There are several methods for synthesis of metal alkanoates including metathesis reactions [16], ligand exchange reactions [17], and anodic dissolution of metal in alkanoic acid. In this study the reaction of Cobalt nitrate, iron sulphate, copper sulphate and calcium stearate in the presence of KOH solution was examined.

***Table 2.*** *Property of palm kernel oil.*

| Property | Values |
|---|---|
| Saponification Value ($cm^3$) | 23.84 |
| Acid Value | 5.46 |
| Free fatty acid | 9.2% |
| Iodine Value | 18.53 |
| pH | 6.70 |
| Viscosity ($Kgm^{-1}s^{-1}$) | 19.62 |
| Moisture content | 1.02 |
| Relative density | 0.911 |

The properties of metal soap are highlighted in Table 3. The pH of the metal soaps varied between 6.3 - 8.1. Co-PKO, Fe-PKO and Cu-PKO were acidic while Ca - PKO was basic. Evaluation of drier production method has reviewed that solvent process of producing metal soap yielded a homogeneous product. This is probably due to lack of interfering by products as result, no problem was encountered during the synthesis of the insoluble salt. The soaps of the alkaline-earth metals were white while those of transition metals were colored. The metal soaps were insoluble in water as a result does not foam well in water. This is an indication that metal soap has poor detergency power as a result of their insolubility in water. The higher the foam stability, the more the ability of metal soap to have detergency property. Even though metal soaps have been confirmed to be insoluble in water but they are soluble in organic solvent such as alkyl benzene sulphonate or lauryl sulphate [18]. These are products that could boost the foaming capacity of soaps and are good components of fire extinguishing agent. Given the properties of the metal soaps (Table 3) it is evident that prepared metal soap is slightly alkaline having pH values ranging from 7.3-8.1. The specific gravity of the calcium soap was found to be higher than those of copper, cobalt and iron metal soap. From the present study, the heavy metal soap (Cu-PKO, Co-PKO, Fe-PKO) has lower specific gravity than titanium dioxide. Interestingly, lower specific gravity of metal soap of heavy metals leads to a considerable cost saving in coatings [19]. The refractive indices of the prepared metal soap were determined using an Abbe's refractometer (Table 3). This illustrates that the heavy metal soaps have refractive index lower than that of calcium soap. This is an indication they could be considered to impart opacity to polymer coatings due to low value of specific gravity. The metal soap oil absorption properties were determined using the spatula rub-out technique and results obtained are shown in Table 3. The various prepared metal soap exhibited roughly the same oil absorption values, and which is higher than the oil absorption calcium soap. Less oil absorption indicates less resin demand without compromising other coating properties [20]. The oil absorption depends mainly on the physical structure (i.e. size and shape of the extender particles), which also affects several other coating properties including flow characteristics, setting tendency, film durability [21].

***Table 3.*** *Properties of metallic soap.*

| Metal Soap | Color | pH | Specific. gravity g/cm3 | Refractive. index | Oil absorption/100gin water | Yield | Solubility | Foam |
|---|---|---|---|---|---|---|---|---|
| Calcium stearate | white | 7.3 | 7.1 | 4.5 | 46 | High | Insoluble | Low |
| Copper sulphate | light blue | 7.7 | 3.2 | 2.68 | 52 | High | Insoluble | Low |
| Cobalt nitrate | purple | 7.5 | 3.0 | 2.71 | 51 | High | Insoluble | Low |
| Iron sulphate | dark brown | 8.1 | 3.2 | 2.70 | 53 | High | Insoluble | Low |

The specific gravity values of paint with metal soap and paint without metal soap (PKO Soap) are shown in Table 4. The table shows that the PKO Soap paint has the highest specific gravity when compared to those formulated using the metal soap. Heavy metal soap can be incorporated into the paints which is considerable cost saving when compared to the incorporation of PKO soap in paint. Specific gravity of paints is used to determine the coverage of paint products on a substrate, that is, the volume occupied by a known weight of paint product [22]. From this study, it is obvious that the values of specific gravity exhibited by the different metal soap are almost uniform (1.45-1.47) with PKO soap higher (1.68). PKO soap formulated paint exhibited the least viscosity value among the metal soap paints studied. This study present the viscosity of paints prepared using metal soap which is observed to decrease as the quantity of PKO soap (paint without metal soap) increased. The range of viscosities observed for the paint samples makes them suitable for application by spray or brush, Tiwari and Saxena [23] who studied fly ash extended coatings obtained viscosities that ranged between 2.0 and 2.58 Pa.s, and noted that the formations were suitable for application by brush or spray. The interaction of various constituents in any coating system determines its viscosity [24]. Viscosity affects the application and flow properties of a coating and is generally adjusted to the intended application [25] [26]. The thickness of a coating is of importance since it profoundly affects the other properties of coatings such as the extent of protection of a substrate by the applied paint. The data obtained on the thickness of the dried paint films in this study are presented in Table 4. The thickness of the paint mix formulated using the metal soap were generally observed to higher than those of PKO soap (without metal soap) formulated paint samples film thickness. According Tan Tian Aik, [27], weight loss due to weathering is proportional to film thickness up to 20 µm, above which the rate of weight loss becomes indifferent of the thickness. Thus, a film thickness of more than 20 µm performs well as a barrier resistant to weathering. The results obtained in this study on the film thickness of the samples studied are in the desired range to be considered for corrosion protection. The drying properties of the prepared paint samples are given in Table 4. The drying properties investigated are dust-free dry, tack-free dry, and dry for recoat and full hardness.

Metal or cation of the metal soap is the active principle which accelerates the oxidation and polymerization reactions associated with the drying oils. Cobalt nitrate, iron sulphate and copper sulphate soaps were used as primary drier while calcium stearate soap (auxiliary drier) which does not promote drying was used in conjunction with the primary driers to accelerate the drying faster. The organic or anion portions of the metal soap used as drier act as carrying or solubilizing agent. The result of drying properties of the paint which include dust free, tact free, dry for recoat, full hardness were promising and a confirmation that the can be substituted as an additive in the formulation of paint. This is also evident in figure 2 where the prime coat (without the metal soap) is matched to the finish coat (with metal soap). The % reflectance of the prime coat were negative which is an indication that the paint need to be amended and result were fairly suitable to be incorporated into the formulation of paint. The drying properties and storage test by the metal soaps is of great value for the production of reliable and high quality paint.

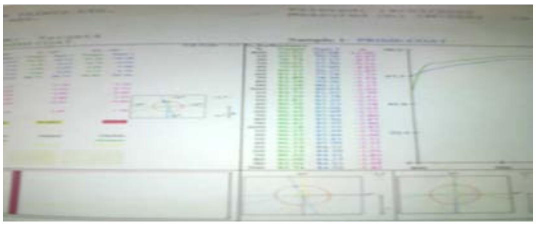

***Figure 1.*** *Reflectance of paint with metal soap.*

The chalking tests carried out on the formulated paint samples indicated that none of the paint samples showed any sign of chalking (figure 2). A good paint is not expected to chalk. The results of the above test showed that the metal soap performed equally well in coatings just like the paint prepared without the metal soap. The formulated paint samples exhibited no sign of skin formation after three months of storage. Various degrees of settling were observed in the samples after three months of storage (Table 5). The settling tests shows that paints formulated using metal soap showed moderate settling tendencies when compared to the formulation without metal soap which almost showed no settling tendency. Though, some properties of metal soap affect their settling tendencies. These include particle size, particle shape, specific gravity, and the activity which includes such properties as basicity, wettability, flocculating, dispersing and gel forming tendencies. The properties of the binder also influence the settling tendencies. Given the settling tendencies of the formulated paints, it is possible that the difference in settling tendency observed for the metal soap paint and

PKO soap formulated paint samples may be due to the difference in their particle structure. Furthermore, specific gravity is known to be related to the sedimentation and granulation of paints and affects the rate at which solid particles settle in liquids. The proportional difference between the specific gravities of these meal soap have led to the slight settling noticed in the metal soap based paint formulations. Oil absorption of pigments also relates to its settling tendencies. Pigments with high oil absorption will tend to absorb the paint vehicle and form coat after an extended period of storage.

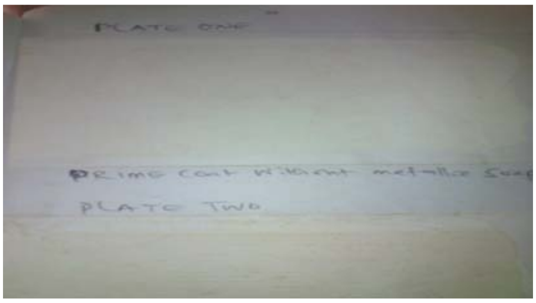

***Figure 2.*** *Prime coat and finish coat.*

***Table 4.*** *Coating properties of prepared metal soap paint.*

| Formulation | code | Specific. gravity g/cm3 | Viscosity | Dry thickness (mm) | film | Color | Drying time tack free dust free dry for recoat | Full hardness |
|---|---|---|---|---|---|---|---|---|
| PKO | 1.68 | 58.00 | 0.20 | 70 | 82 | White | 170 | 0.46 |
| Cu-PKO | 1.47 | 71.00 | 0.32 | 66 | 83 | Green | 150 | 0.38 |
| Co-PKO | 1.45 | 70.13 | 0.32 | 66 | 71 | Yellow | 150 | 0.42 |
| Fe- PKO | 1.46 | 68.18 | 0.31 | 65 | 70 | Black | 140 | 0.41 |

From Table 5, the performance of the formulated paints shows that all the metal soap paint samples exhibited good adhesion properties. All the prepared paint samples exhibited % adhesion loss in the range, 1 - 10. Adhesion according to ASTM D 907 (2012) is defined as the state in which two surfaces are held together by interfacial forces which may consist of valence forces or interlocking action or both. For the most part, organic coatings are removed in service by abrasion, chipping, coins, and other instruments, picking away at exposed edges, corrosion of the substrate, impact or impingement by stones. All the formulated metal soap paint samples did not show any sign of mildew formation after two months of exposure to rain, and sunlight. This is an indication that the PKO soap paint performed equally well in coatings just like the metal soap formulated paint samples. On the other hand, the alkyd paint formulated samples shaded their color after twelve months of exposure to rain and sunlight. The paint formulations containing PKO Soap exhibited slight rust while all the metal soap based paint formulations resisted rust. This is an indication that both soap formulations can withstand environmental challenges, and therefore can be used in corrosive environment.

***Table 5.*** *Storage test of prepared metal soap paint.*

| Formulation | code | Adherence Test % | Settling test | Chalking | Mildew formation | Skinning | Gloss Rain water | Media resistance sunlight |
|---|---|---|---|---|---|---|---|---|
| PKO | 8 | 8 | None | None | None | Mat | 0 | 0 |
| Cu-PKO | 6 | 6 | None | None | None | Mat | 0 | 0 |
| Co-PKO | 6 | 6 | None | None | None | Mat | 0 | 0 |
| Fe- PKO | 6 | 4 | None | None | None | Mat | 0 | 0 |

***Table 6.*** *Rating on degree of settling of paint.*

| | Rating Description of paint condition |
|---|---|
| 10 | Perfect suspension, no changes from original condition of paint |
| 8 | A definite feel of settling and a slight deposit brought on spatula |
| 6 | Definite cake of settled pigments, spatula drops through cake to bottom of the container under its own weight |
| 4 | Spatula does not fall to bottom of container under its own weight. Difficult to move spatulathrough cake sidewise and slight edge resistance |
| 2 | Definite edgewise resistance to movement of spatula |
| 0 | Cake that cannot be reincorporated with the liquid to form smooth paint by stirring manually |

# 4. Conclusion

In this study, the oil were evaluated and used to prepare Copper, Iron, Cobalt and Calcium soap of Palm Kernel Oil by metathesis in aqueous alcohol. Metal soaps from PKO was employed as driers in paint and can be equally applied in varnish, printing ink and linoleum industries to accelerate the change of the liquid oil or synthetic vehicles to elastic solid. Also, the results provide additional impetus for developing the performance of industrially useful products of Palm Kernel Oil. The properties of the formulated metal soap paint were promising with strong insight to a secured future. This is likely as they are expected to find utilization in the surface coating industry which will lead to reduction on over dependence on imported drier in paint, thereby creating jobs as well as save scarce resources.

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