Emulsions Preparation from Esterified Waste Polyethene Waxes and their Applications on Wood and Leather Substrates

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Abstracts: Esterified waxes were compounded with toluene, oleic acid, morpholine and distilled water to produce the emulsions. The emulsions produced were applied on wood and leather, which adhered and dried. The coated substrates showed decrease in water absorption coefficients compared to the uncoated surfaces. The coated leather showed better marking-off properties than the wood coated substrates. The emulsions applied on wood finishing show good absorption properties which can be used as undercoat emulsion for wood.

Keywords: emulsions, esterified waxes, marking-off properties, water absorption coefficients.

I. Introduction

Polyethene (PE) is the most commonly used commodity in the environment and one of the commonest wastes that are found across all communities in Nigeria. The increasing menace of waste PE littering the environment causes environmental pollution [1]. Based on the report from the Federal Environmental Protection Agency (FEPA); a bulk of the refuse dumps in Nigerian environment are PE bags and other plastics [2]. Polyethene products are non-biodegradable and they constitute health and environmental hazards because they encourage the spread of water borne diseases, constitutes dirty environment and can cause suffocation to small children and animals [3]. Control of environmental pollution by waste recycling is the best method of reducing bulk of the residue requiring disposal. Waste PE recycling is an act of treating waste PE materials like used water sachets and others waste PE in such a way they can be used again. This research work is justified in that it aims at minimizing the amount of waste PE in the environment by adding value to the waste for useful domestic purposes, such as emulsion.

Polyethene as the most commonly used polyolefin has no polar functional groups in its backbone to provide reactive sites [4, 5]. Polyolefin modified by the introduction of polar groups in their backbones improves the physicochemical properties of the parent polymers and promotes adhesion, dyeability, cross-linking, or other chemical modifications [4, 6, 7, 8, and 9]. Polyethene waxes modified with maleic anhydride and subsequently reacted with polyhydric alcohols are found to be suitable for the preparation of emulsion [10].

An emulsion is a thin light-sensitive coating on a medium such as gelatin on film. It has been necessary from the view point of appearance and protection to repeatedly polish articles such as the floors of building made from tiles, plastics, etc., furniture, metals and plastics; the outer surface of car bodies; and the likes [10].

Many materials absorb moisture when immersed in water or when they are placed in a humid atmosphere [11]. The study mentioned that understanding water absorption by substrates during soaking is of practical importance, since it affects the mechanical properties of the product. He added that all strength properties decrease as substrates absorb moisture in the hygroscopic range. Khazaei also stated that one of the most important aspects of substrates technology is modelling of the water absorption processes [11].

Marking-off test of coated substrates is a process of assessing the resistance of coated substrates to marking-off. According to B.S. 3662/5 marking-off test standard, the coated substrates are stored under heat and pressure in contact with filter paper. The assessment of the staining of the filter paper is done with the standard Grey Scale for assessing stain. Substrates are applied on their surfaces some form of coatings to impart weathering protection, decoration, etc. [12].

II. Materials And Methods

2.1 materials

The equipment used in the study include of a Nicolet-IR100 FT-IR spectrophotometer, test tubes, beakers, and conical flasks. Others are volumetric flasks, measuring cylinders, and spatula.

Modified waste polyethene waxes [13] esterified with ethylene glycol and glycerol reported elsewhere [14] were used; P200S1EE, P200S2EE, P200S3NEE and P250S1NEE were esterified with ethylene glycol and P200S1EG, P200S2EG, P200S3NEG and P250S1NEG esterified with glycerol respectively.
2.2 Methods

The esterified waxes prepared elsewhere [14] was compounded with other ingredients at room temperature to produce the emulsions. The following formulation was used for the preparation.

<table>
<thead>
<tr>
<th>Table 1. Weight Percentage of Ingredients used in Emulsion Preparation</th>
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<tbody>
<tr>
<td>Materials</td>
</tr>
<tr>
<td>-------------------</td>
</tr>
<tr>
<td>Esterified wax</td>
</tr>
<tr>
<td>Toluene</td>
</tr>
<tr>
<td>Oleic acid</td>
</tr>
<tr>
<td>Morpholine</td>
</tr>
<tr>
<td>Water</td>
</tr>
</tbody>
</table>

The esterified waxes were dissolved in toluene and then oleic acid, morpholine and the distilled water were added respectively with continuous stirring.

2.3 Infrared Spectroscopy

The Infrared spectroscopy analysis of the waxes and the emulsions was determined using KBr method on Nicolet-IR100 FTIR spectrophotometer. The technique is capable of detecting specific functional groups in polymers thereby helping in their identification and characterization [2].

2.4 Coating of Substrates

The prepared emulsions were applied onto wood and leather using brush, as presented on Plate 2.

2.5 Water Absorption Test of Substrates

The water absorption coefficients determination give an idea of the water uptake of the coated substrates compared to the uncoated ones. The ability of the coated substrates to resist water uptake compared to the uncoated substrates can then be observe.

Substrates were cut to approximate dimensions of 5 x 2.5 cm². One face of the substrates was used for test. The other sides were coated with wax to stop water uptake through their sides. The initial moisture contents of substrates were obtained. Water absorption data were obtained by placing the substrates in 2 litre screw-cap flask containing distilled water. Tests were conducted at 25 ± 0.5 °C for immersion periods ranging from 30 minutes to 24 hours. After soaking, the moisture content of substrates was calculated based on the increase in the substrate weight at corresponding times. At regular time intervals, ranging from 30 minutes at the beginning to 3 hours during the last stages of the process, the substrates were rapidly removed from the flask and superficially dried on a large filter paper to eliminate the surface water. The substrates were then weighed to determine the moisture uptake. The substrates were subsequently returned into water and the process was then repeated.

Finally, curves showing the cumulative weight gain versus the square root of time were plotted, and linear regression curves were computed for each substrate. The water absorption coefficients of the substrates were determined by using Equation 1: [11]

\[ m_w = A \sqrt{t} \]  

Equation 1

Where, \( m_w \) is the amount of water absorbed in kg/m², and \( A \) is the water absorption coefficient (kg/m² s\(^{1/2}\)). Following the definition, the water absorption coefficient, \( A \) is given by the slope of the fitted curve divided by the contact area.

The water absorption coefficients for the coated substrates were then calculated and recorded; the results are shown in Figure 1.

2.6 Marking-off Test of Coated Substrates

This method is intended for assessing the resistance of the coated substrates to marking-off. A piece of filter paper 5cm x 5cm was placed on each of the coated substrates 5cm x 4cm so that an area of 5cm x 1cm of the filter paper was not in contact with the coated substrates. The substrates were covered with a glass plate 5cm x 5cm and a weight of 1Kilogram was placed on the glass plate. The substrates were kept in the oven at 60 °C for 4 hours. This is B.S. 3662/5 marking-off test standard. The staining of the filter papers was assessed with the standard Grey Scale for assessing stain. The assessment were observed and recorded as shown in Figure 2.
III. Results And Discussion

The prepared emulsions were shown in Plate 1.

P200S1EE Emulsion
P200S1EG Emulsion
P200S2EE Emulsion
P200S2EG Emulsion
P200S3NEE Emulsion
P200S3NEG Emulsion
P250S1NEE Emulsion
P250S1NEG Emulsion

Plate 1. Prepared Emulsions

3.1 infrared spectra

The functional groups of the esterified waxes and the emulsions were identified from their infrared spectra. The summary of the functional groups of the waxes and the emulsions are presented on Table 2. The infrared spectra are used to identify the functional groups in a compound. Table 2 presented the functional groups of the waxes and the emulsions as observed from its spectra in the appendix (Figures 3-18). Spectra of waxes esterified with ethylene glycol (Figures 3-6), indicate bands for C-H stretch (aliphatic), C=O stretch (acids), C-H bend (aliphatic) and C-H bend (alkene). Combination of O-H stretch and C-O stretch around 2290 cm\(^{-1}\), O-H stretch (H-bonded) first overtones around 1600 cm\(^{-1}\) and O-H bend around 1373 cm\(^{-1}\) were also observed. The presence of O-H bands indicates the evidence of esterification reaction between the modified PE waxes and the alcohols [14]. The spectra of waxes esterified with glycerol (Figures 7-10), in addition to the bands mentioned above were O-H (H-bonded) above 3360 cm\(^{-1}\), C-O stretch above 1040 cm\(^{-1}\) and O-H bend association around 2356 cm\(^{-1}\). This justified that glycerol is trihydric alcohol having more O-H bands compared to ethylene glycol which is dihydric with less O-H bands.

Table 2. Infrared Spectra analysis of the Waxes and the Emulsions

<table>
<thead>
<tr>
<th>Peaks (nm)</th>
<th>Assignment</th>
<th>Peaks (nm)</th>
<th>Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Above 3360 (broad)</td>
<td>O-H stretch (H-bonded)</td>
<td>Above 3600 (sharp)</td>
<td>O-H stretch (free)</td>
</tr>
<tr>
<td>2853-2924</td>
<td>C-H aliphatic stretch</td>
<td>Above 3400 (weak)</td>
<td>N-H stretch (I° amines)</td>
</tr>
<tr>
<td>Around 2500</td>
<td>O-H stretch (overlap)</td>
<td>Around 2507</td>
<td>O-H stretch/O-H bend (combination)</td>
</tr>
<tr>
<td>Around 2356</td>
<td>O-H bends (association)</td>
<td>Above 2100</td>
<td>O-H stretch plus C-O stretch</td>
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</tbody>
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Emulsions Preparation from Esterified Waste Polyethene Waxes and their Applications on Wood…

<table>
<thead>
<tr>
<th>Frequency (cm⁻¹)</th>
<th>Description</th>
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<tbody>
<tr>
<td>2290</td>
<td>O-H stretch &amp; C-O stretch (combination)</td>
</tr>
<tr>
<td>1706-1718</td>
<td>C=O stretch (acids)</td>
</tr>
<tr>
<td>1599-1601</td>
<td>O-H stretch H-bonded (1ˢᵗ overtones)</td>
</tr>
<tr>
<td>1457-1461</td>
<td>C-H bend (aliphatic)</td>
</tr>
<tr>
<td>1373</td>
<td>O-H bend</td>
</tr>
<tr>
<td>1304</td>
<td>C=O stretch</td>
</tr>
<tr>
<td>1295</td>
<td>C-N stretch (amines)</td>
</tr>
<tr>
<td>1044</td>
<td>C-O stretch</td>
</tr>
<tr>
<td>724</td>
<td>C-H bend (alkene)</td>
</tr>
</tbody>
</table>

In addition to the bands observed on spectra of the esterified waxes, the spectra of the emulsions (Figures 11-18) showed O-H stretch (free) above 3600 cm⁻¹, N-H stretch primary amine above 3400 cm⁻¹, O-H bend at 2091 cm⁻¹, N-H bend (amine) around 1650 cm⁻¹, C=C stretch (aromatic ring) around 1552 cm⁻¹, C-N stretch (amine) around 1313 cm⁻¹ and C-H (out-of-plane bending) and ring puckering or N-H wagging on H-bonding at 869-880 cm⁻¹. Combination bands are also observed for O-H stretch plus O-H bends around 2507 cm⁻¹ and O-H stretch plus C-O stretch above 2100 cm⁻¹. The appearance of bands for N-H stretch, N-H bends and C-N stretch (amines) is evidence of the reaction of the esterified waxes and morpholine. Bands of C=C stretch (aromatic rings) around 1552 cm⁻¹ indicates presence of toluene. Presence of more O-H stretch (free) and combinations bands for O-H stretch plus O-H bends indicates presence of oleic acid and water used in emulsions preparation.

3.2 Coating of Substrates

The emulsions (Plate 1) applied on wood and leather substrates respectively were dried as showed in Plates 2.
3.3 Water Absorption of Substrates

The water absorption of the uncoated and coated substrates was calculated using the slopes on plots of cumulative water gain with t^{1/2} of wood and leather coated with the emulsions. The calculated water absorption coefficients A, of the coated and the uncoated substrates were recorded as shown in Figure 1.

Figure 1 showed decrease in water absorption coefficient of the coated substrates compared to the uncoated substrates. Understanding water absorption by substrates during soaking is of practical importance, since it affects the mechanical properties of the product [11]. Moisture can decrease the strength properties of the substrates with increase in water/moisture absorption when exposed to the environment during usage. Microorganisms might also act on the moist substrate, thereby damaging it. The decrease in water absorption coefficient of the coated substrates over uncoated suggests increased strength properties. All strength properties decrease as substrate adsorbs moisture in the hygroscopic range [11]. This means that the emulsion prepared can be used on wood and leather since it reduced its water absorption.
3.4 Marking-off of Coated Substrates

The marking-off of the coated substrates was assessed using the Standard Grey Scale for assessing stain. Figure 2 showed the marking-off assessment of the coated substrates observed. The marking-off assessment of wood substrates coated with P200S1EG, P200S1EE, P250S1NEE, P250S1NEG, and P200S3NEG are relatively poor having an assessment of 2, 2/3 and 3 respectively. Whereas P200S3NEE, P200S2EG and P200S2EE gave relatively fair results with stain assessment of 3/4 and 4 respectively, which are good. The assessment of the marking-off of leather coated substrates is 4/5 to 5 which shows a very good marking-off assessment. This shows that the emulsions have a better marking-off on leather compared to wood. This suggests that the emulsions prepared are suitable on leather as polishing emulsions. According to Vipond, regardless of the purpose of the substrate, maintenance may involve ensuring that the substrate is preserved as intended [12].

![Marking-off assessment graph](image)

Figure 2. Marking-off Assessment of Coated Substrates

IV. Conclusion

Waste polyethylene waxes esterified with two different polyhydric alcohols are compounded into emulsions. The emulsions produced were applied on wood and leather to which they adhered and dried. The coated substrates showed decrease in water absorption coefficients compared to the uncoated ones. The coated leather showed better marking-off assessment compared to the coated wood finishing.

The emulsions prepared can be used as undercoat in wood finishing which will effectively reduce the amount of water absorption by the wood. They can also be used on leather as polishing emulsion, having good marking-off properties. Thus, the production of emulsion from modified waste PE waxes reduces environmental pollution and recovers valuable by-products (emulsions). It also serves as a means of creating job opportunities for the people.

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References


Emulsions Preparation from Esterified Waste Polyethylene Waxes and their Applications on Wood…


APPENDIX

Infrared Spectra

Figure 3. P200S1EE Wax

Figure 4. P200S2EE Wax
Figure 5. P200S3NEE Wax

Figure 6. P250S1NEE Wax

Figure 7. P200S1EG Wax

Figure 8. P200S2EG Wax
Figure 9. P200S3NEG Wax

Figure 10. P250S1NEG Wax

Figure 11. P200S1EE Emulsion

Figure 12. P200S2EE Emulsion
Emulsions Preparation from Esterified Waste Polyethylene Waxes and their Applications on Wood...

Figure 17. P200S3NEG Emulsion

Figure 18. P250S1NEG Emulsion