

Comparison of Physical and Chemical Properties, and Applicability of Polyurethane Industrial Coating Systems Synthesized from Multiple Cooked Coconut Oil vs. Fresh Coconut Oil

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ABSTRACT

There is a potential health risk to continuous use or disposal into the animal feed of several times cooked plant oils. Therefore, the experiment turned them into a useful eco-friendly substitute in the paint industry to replace the main crude oil born polyol in the polyurethane (PU) coating system to keep the lowest contribution in global warming. Two coconut oil-derived bio-based polyols (BBP) were synthesized using fresh coconut oil and waste coconut oils. The acid value, viscosity, reaction water release, oil length, FTIR spectrum, and Lovibond colorimetric index were assessed during the synthesis. Three series of white-pigmented polyurethane paint coatings were prepared using fresh bio-based polyol (FBBP) by fresh coconut oil, waste bio-based polyol (WBBP) by waste coconut oil, and commercially available polyol (CAP). Physical and chemical properties such as fineness of grinding, viscosity, film hardness, flexibility, impact resistance, density, gloss, touch drying time, hard drying time, adhesion, dry film thickness, water resistance, solid content, and colour deviation were tested among them. According to the results, it was proven that both PU series of BBP equally performed in many physical However, PU with WBBPs was better in drying time and PU with FBBPs were better in viscosity and colour deviation. When both series compared PU with CAP, it was proven that all three series were equally performed in film hardness, adhesion, water resistance, flexibility, and impact resistance. But both PU series of BBP were better in drying time and PU series with CAP was better in density, gloss, dry film thickness, and solid content

Keywords: RBD coconut oil, waste coconut oil, renewable polyurethane, physical properties.

INTRODUCTION

Several times cooked plant oils badly affect human health. Heated oils perform free radicals which can increase the oxidative stress to attach human tissue in cellular and molecular levels. High peroxide values (PV) found in heated oils in chemical and histopathological observations showed potential damage to the jejunum, colon, and liver in tested animals using 3 times repeated cooking oils [1]. Some studies showed intense risk of hypertension and cancers in the prostate, breast, esophagus, and larynx [2]. Used cooking oil can be further used when it does not exceed mentioned values such as FFA 1%, PV 10 m eq/kg, total polar compounds (TPC) 15.5. TPC is another parameter that is widely used in Europe to assess used plant oil [3]. Those days waste cooking oils were used to produce animal feed but eventually found that some hazardous substances transmitted forward along food chains. Therefore, doors opened to find new alternative disposal methods for used cooking oils. As a result, many valueadded products were developed in many locations in the world [4].

Chemical changes associated in waste cooking oils

Heated oils undergo hydrolysis reaction with moisture in foodstuff to cause breakage of the ester bond. As a result, it forms free fatty acids (FFA), monoacylglycerols, and diacylglycerols. Air and high temperature facilitate oxidation and thermal alteration of unsaturated sites in fatty acids and undergo free radical chain reactions. Oxidation results in oxidized monomers, dimers, oligomers, polymers, and volatile compounds such as aldehydes, ketones, alcohols, and hydrocarbons. Thermal alterations give cyclic monomers, trans monomers, non-oxidized dimers, and oligomers [2].

Purifying methods apply for used oils

Changes in colour, odour, and FFA can be reduced by activated carbon in waste cooking oil (WCO) [5]. Using adsorbents like rice, potato, and charcoal to precipitate impure materials is the community's general practice in purifying WCO. But active charcoal is a product that can be used to remove anions, cations, inorganic molecules, and organic molecules in solution or gas. According to the experiment, 5.5 grams of active charcoal in 50 grams of WCO for 80 minutes adsorbed up to 97.91% [6]. Salak peels, coconut shells, and banana peels produce activated charcoal. Coconut shells and banana peels adsorb free fatty acid content of 33.71% and 52.73% accordingly in WCO [7]. Sliced and crushed fresh ginger also can be used for the filtration of WCO to some extent [8]. Paper filters can remove 78.63% of solid wastes in WCO. Silica gel can be used to remove moisture and 0.11 g of silica gel per millilitre of oil at $15C⁰$ temperature showed good efficiency [9]. Normally WCO contains 10-20% solid materials depending on the cooking process. Very firstly fabric bags or stainless steel meshes around $150-200\mu$ m under 60-65[°]C temperature can be used to remove solid materials.

Role of coconut oil in the paint industry

Virgin coconut oil is a good starting material for producing lacquer enamel paints and clear gloss lacquer enamels while deriving it into useful binders such as alkyd resins. Virgin coconut oil is an unrefined oil taken from fresh coconuts. But normal coconut oil coming under RBD grade (refined, bleached, deodorized) originates from the dried kernel called copra [11]. Concerning health and environmental impacts, a trend has been created to use more green products. Coconut oil-derived alkyd resins can be used to replace hazardous fossil-originated plasticizers in nitrocellulose surface coatings. Plasticizers prevent paint films from cracking by mechanical forces and the non-drying nature of coconut oil supports the plasticizing [12]. Industrial coating systems follow three coat systems which start from the bottom to the top on metal substrates such as metal primer, topcoat paint, and clear lacquer. But direct-to-metal (DTM) paint is used as a one single paint coat which directly applies on metals. Coconut oil derived polyols can be used to prepare DTM coatings under polyurethane chemistry [13]. PU is one of the versatile coating systems in automotive, furniture, construction, and footwear, because of its high performance. The PU film cures gradually during the completion of the reaction between polyols and polyisocyanates. Coconut oil-modified polyols can give promising results in PU anticorrosive metal primers even without anticorrosive pigments [14].

The main intentions of this research are to design, synthesize, characterization of bio-based polyols (BBP) using purified waste coconut oil and fresh coconut oil. And preparation of separate two PU coating systems of them to evaluate physical and chemical properties relative to crude oil-derived PU coating systems with commercially available polyol. Comparatively tested BBP (alkyd resin) in the reference [12] represents the main backbone of the bio-based PU system.

MATERIALS AND METHODS

Refined bleached deodorized coconut oil from Sena mills in Sri Lanka, three times fried domestically collected coconut oil, phthalic anhydride, benzoic acid, pentaerythritol, glycerine, xylene, and lithium hydroxide as a catalyst used for synthesis FBBP and WBBP. Butyl acetate and titanium dioxide 80-99% pigment was obtained by Millennium Chemicals, Australia. Whatman filter papers 125mm diameter Cat No 1001 125 and 200 size mesh were used to filter the WCO.

Lab equipment – Five neck flask with the heating element, condenser and nitrogen supply, body tube, Dispermat lab mill, spray gun, Hegman gauge by Sheen Instruments Ltd, Sheen A15 viscosity flow cup, Sheen specific gravity cup, coating thickness meter by Biuged Instruments BGD 542/2, TQC Sheen polygloss meter, Sheen cylindrical mandrel bend tester, Erichsen GMBH pencil hardness tester, Sheen 750 cross cut tool, 3M scotch tape series 4-1000, Datacolour 200R spectrophotometer, PerkinElmer Spectrum Two FTIR, NICOLET, Model - 380 FTIR- Thermo electron Corporation, Sheen impact tester.

A. Designing formulations for BBPs

The modified formulation of the previous study [14] was followed to synthesize BBP using RBD coconut oil

and WCO separately. BBPs which were synthesized by RBD coconut oil and WCO are named accordingly FBBP, WBBP.

Table I. Theoretically Designed Formulations Of Bbp Made With Coconut Oils [12]

B. Filtering of WCO

5% Brine aqueous solution was prepared and added 50% by weight relative to waste coconut oil. The mixture was heated up to 50° C for 1 hour while stirring in a magneto stirrer at speed 5. The brine solution layer with water-soluble impurities was removed physically and the separated WCO layer was heated to 110^0 C for 15 minutes to remove moisture. The WCO was filtered using a 200-mesh followed by Whatman filter paper.

C. Synthesis of BBPs

The following raw materials were loaded into the five-neck reactor vessel and heated up to 250^0 C while stirring at 100 rpm. The mixture was kept for 0.5 hour in a nitrogen gas environment. A methanol verification test was conducted, and a clear uniform solution was obtained It was confirmed the completion of the monoglyceride stage.

Upon cooling the reaction vessel to 120^0 C temperature, the following raw materials were introduced to the vessel.

Heat the mixture while stirring and 6 samples were taken in 45 min equal intervals to measure viscosity, acid value, and reaction water release. BBPs were synthesized according to the above formulation.

D. Millbase preparation of PU wet samples

The mill base (pigment dispersion) was prepared by "Dispermat", Type 6DU2-547 (Germany) mill, and a dispersion blade with 6 cm diameter Teflon was used for dispersion. 1 mm in diameter of zirconium beads were used in 2:1 ratio by weight (mill base: beads) under 4000 rpm, 30^0 C, and 40 min for the preparation. All the other materials were added according to the following tables and stirred at 2000rpm for 10 minutes for completion of the wet samples.

Table II. Common Composition of Millbase Dispersion

Separate millbases were prepared for all PU wet samples.

E. Preparation of PU wet samples

1) PU wet samples which were prepared by commercially available crude oil-originated polyol

Table III. Formulations of Pu Wet Samples Prepared By Cap In Weight %

2) PU wet samples which were prepared by FBBP

Table IV. Formulations Of Pu Wet Saples Prepared By Fbbp In Weight %

3) PU wet samples which were prepared by WBBP

Table V. Formulations Of Pu Wet Samples Prepared By Wbbp In Weight %

F. Spray application

Machine - Panels applied under 29° C, relative humidity 88% environment. Maintained 3.5 bar gun air pressure, a Devilbiss JGA 54 (England) spray gun with a 1.3 mm nozzle diameter was used for spray applications.

Common dilution was followed for all PU samples. The dilution ratio between PU paint samples and hardener was 2:1 ratio. AkzoNobel, Sikkens P25 hardener was used as the hardener for PU paints.

Two single spray paint passes were applied on steel panels with the following specifications.

Q-panels -150mm x 50mm x 0.6mm.

Bend panels – 150mm x 50mm x 0.6mm (use only for cylindrical mandrel bending test)

Metal surfaces of all panels were prepared according to ASTM 1731-67.

G. Testing

1) Determination of free fatty acid (FFA) content in coconut oil: ASTM D 5555-95 (2001) was followed to measure the FFA of coconut oil which is used to synthesize BBPs.

2) Determination of acid value of synthesized BBPs: ASTM D 1541-86 was followed to measure acid values of BBP during the synthesis.

3) Methanol verification test of monoglyceride: 1.0 ml of the monoglyceride reaction mixture in the reactor was taken out and 1.5 ml of methanol was added to the test tube at room temperature. Uniformity was observed just after stirring and the uniform mixture confirmed the completion of the monoglyceride stage.

4) Measuring viscosity of BBP samples during the synthesizing process: the synthesizing resin sample was taken out from the reactor and a calculated amount of xylene was added to the non-volatile mixture to become a resin. The resin was poured into a body tube and a small air gap was kept inside the body tube. Time taken was measured to travel air bubbles from bottom to top in the body tube as the viscosity at 25° C.

5) Determination of oil length of BBP: Oil length is expressed as the weight percentage of glyceride oil in nonvolatile in an alkyd resin.

Oil length = $(W \times 100) / (NV - W_R)$

 $W = Weight of glyceride oil$

 $NV = Weight of non-volation$

 W_R = Weight of reaction water

6) Fourier Transform Infrared Spectroscopy (FTIR): Functional groups in BBP samples and coconut oil were determined by NICOLET, Model - 380 FTIR, Thermo electron Corporation, and Spectrum Two PerkinElmer FTIR, relevant spectrographs were taken under $400 \text{cm}^{-1} - 4000 \text{cm}^{-1}$ range.

7) Pencil hardness test of a dried paint film: ASTM D 3363-74 was followed and measured resistance to scratch of PU paint coatings which were cured for 7 days. Erichsen pencil hardness tester was used.

8) Cylindrical mandrel bending test for dried paint film: ASTM D 1737-62 test method was followed. A 2mm spindle is used to assess the bending of PU paint coatings which were cured for 7 days.

9) Impact resistance test for a dried paint film: ASTM D 2794-84 test method was followed. 500g weight had fallen down from 3 feet height and several cracks were observed on dried PU paint films which were cured for 7 days. 5 impacts were made and cracks were observed by a magnifying glass.

10) Density of paint: The ASTM D 1475 test method was followed for wet PU paint samples.

11) Colorimetric value of varnish by Lovibond Tintometer: BS 684 test method was followed to determine colour values of BBP and coconut oil.

12) Spectrophotometry: The ISO 7724-1, Paints and varnishes -Colourimetry. Colour differences among colours are projected into a spherical coordinate system according to CIE Lab1964 by using Datacolour 200R computerbased spectrophotometer. Total deviation expressed using CIE DE value. When it increases colour deviation relative to standard colour increases. In this experiment, dry panels of PU paints with CAPs were used as standard panels, and colour deviation was measured relative to dry panels of PU paints with BBPs. Three sets of dried PU paint panels which were cured for 7 days and with equal concentrations of polyols as 45%, 55%, and 65% were tested.

13) Apparent viscosity: The BSA15 flow cup was used. The time taken to fill up a 50cc cup through the BSA15 by wet PU paint samples was measured.

14) Dried film gloss: ASTM D523-80 test method was followed. The gloss value of PU paint coatings was measured after 48 hours of drying period by 60^0 angles.

15) Touch drying time of a paint film: Time is taken to obtain no permanent finger marks at a light touch on applied PU paint films.

16) Hard drying time of a paint film: Time taken to obtain no permanent finger marks at heavy touch applied on PU paint films.

17) Crosshatch test: The ASTM D 3359-83 test method was followed to assess dried PU paint coatings and the below ratings were given. The evaluation rating table can be referred from the previous [12], [13], [14] works.

18) Dry film thickness of a paint film: The ASTM D 1186-81 test method was followed to measure the thickness of dried PU paint coatings.

19) Water immersion test of organic coatings on steel: ASTM D 870-54 test method was followed. Painted panels were kept for 7 days and submerged in distilled water for 18 hrs. The appearance of the submerged and the rest of the panel was compared for wrinkles, chalking, blisters, or any specified.

20) Fineness of grinding of a wet paint sample: The ASTM D 1210 test method was followed to assess the millbase of wet PU paint samples to maintain particle size 0-5µm.

21) Solid content: The SLS 489:1980 test method was followed to assess wet PU paint samples.

RESULTS

A. Characterization of coconut oils

Table VI. Physical Properties Of Coconut Oils

4*. FTIR spectrum:*

Fig 2. FT-IR of waste coconut oil

B. Characterization of BBPs

1. *FTIR spectrum:*

Fig 3. FT-IR of synthesized FBBP at the 6th sample

Fig 4. FT-IR of synthesized WBBP at the $6th$ sample

2. Viscosity of BBPs by the body tube during the synthesis: The first sample was drawn after 30 minutes of reflux begun. Continuously 6 samples were drawn while keeping 45 minute intervals.

Fig 5. Viscosity changes during the synthesis of BBPs.

4. Reaction water collection of BBPs during the synthesis: The first sample was drawn after 45 minutes of reflux. Continuously 6 samples were drawn while keeping 45-minute intervals.

Fig 6. Total collection of reaction water of BBPs during the synthesis

Fig 7. Collected reaction water of BBP in 45-minute intervals.

5. Acid values of BBP during the synthesis: The first sample was drawn after 45 minutes of reflux. Continuously 6 samples were drawn while keeping 45 minute intervals.

6. Properties of Polyols

Table VII. Property comparison of polyols

Coconut oil **FBBP** Waste coconut oil WBBP

Fig 9. Appearance of coconut oils and synthesized BBPs

C. Physical properties of wet PU paint samples

1. Fineness of grinding by Hagman gauge: All PU paint samples were maintained 0-5µm particle size.

2. Solid content (%) of all PU paint samples:

Fig 10. Variation of the solid content of PU paint samples

3. Density (Specific gravity) of PU paint samples:

Fig 11. Variation of specific gravity of PU paint samples

Fig 12. Variation of the apparent viscosity of PU paint samples by flow cut BSA15

5. Touch drying time:

Fig 13. Variation of touch drying time of PU paint samples

6. Hard drying time:

Fig 14. Variation of hard drying time of PU paint samples

D. Physical properties of dried metal primer coatings on steel panels (Q panels)

1. Dry film thickness (DFT):

Fig 15. Variation of dry film thickness of PU painted panels

2. Gloss 60⁰

Fig 16. Variation of gloss 60 degrees angle of PU painted panels.

- *3. Pencil Hardness Test:* All specimens were satisfied with 4H pencil hardness.
- *4. Crosshatch test results:* All specimens were satisfied with 5B highest adhesion.
- *5. Water immersion test:* No chalking and no gloss reduction were observed visually in all panels.
- *6. Cylindrical mandrel bending test:* All panels were bent successfully without cracking.
- *7. Impact resistance test:*

Table VIII. Number of Cracks Observed Against Five Impacts

	45/weight%	55 weight %	65 weight %
CAP			╭
FBBP			⌒
WBBP			⌒

8. Spectrophotometer analysis of colour differences

Fig 17. Differences of L, a, b values in 45% polyol containing PU dry panels from left to right accordingly FPPB and WBBP against CAP as the standard panel.

Fig 18. Differences of L, a, b values in 55% polyol containing PU dry panels from left to right accordingly FPPB and WBBP against CAP as the standard panel.

Fig 19. Differences of L, a, b values in 65% polyol containing PU dry panels from left to right accordingly FPPB and WBBP against CAP as the standard panel.

Table IX. Final Colour Deviation Value (De Cie) Of Fbbp And Wbbp Against Cap

Polyol content	45%		55%		65%	
Specimen	FBBP	WBBP	FBBP	WBBP	FBBP	WBBP
DE CIE	0.77	2.00	0.65	2.61	0.55	2.79

DISCUSSION

A) Discussion on BBP synthesis

Following functional groups were identified according to FTIR spectra in both fresh and several times cocked coconut oils

Table X. Suspected Functional Groups In The Ftir Spectra [56]

FT-IR: During the condensation reaction of both BBPs (Fig 3 and Fig 4) has shown a prominent peak at 1727 cm⁻¹ which corresponds to aromatic ring ester. It confirms the condensation reaction during the synthesis of phthalic anhydride and benzoic with monoglyceride and pentaerythritol in the reaction mixture. During the condensation reaction, BBP has shown a remarkable peak at 1122 cm^{-1} and 1119 cm^{-1} which is corresponding of the – ester group. In the IR spectra a particular peak at 2678 cm^{-1} which corresponds to the cis distribution of –C=C– has gradually disappeared. Cis distributed double bonds are only present in unsaturated fatty acids in coconut oil monoglycerides. During the synthesis of BBP, those double bonds have used in any other bond formation. Mainly in crosslink formation between polymer chains.

The acid value, reaction water, and viscosity: According to Fig-8, the acid value of both BBP decreased over time as the acid groups in the mixture were replaced with OH groups to form polyester through a condensation reaction. The viscosity increased as shown in Figure 5, due to the formation of polymer molecules. Figure 7 indicated that at the start of the condensation reaction, there was a high release of water due to the high probability of reaction acid-base groups, but this decreased over time.

Oil length: The polyol was confirmed to be a short oil alkyd resin with a fairly low oil length value.

Colorimetric value by Lovibond tintometer: As shown in Table VII, the Lovibond tintometer value of the FBBP and WBBP accordingly was 8-10 and 45-50. These values representing the physical appearances of BBPs were shown in Figure 9. It was clear physically also that the FBBP was very light yellow transparent but WBBP has appeared very dark brown and a low transparent colour. This excessive yellow tone has a considerable effect on white colours.

B) Discussion on PU wet samples

Solid content and specific gravity: The specific gravity indicates the amount of solid materials present in a wet sample. In practice, there appears to be a relatively equal relationship when comparing Figure 10 and Figure 11. The pigment level was equal in all UP-paint samples, therefore these factors varied with the solid content of BBPs. Table VII showed very close values among BBPs in solid content and it was further confirmed in Figure 10 and Figure 11.

Touch drying time and hard drying time: PU paint samples with WBBP showed very low values but the highest values were shown by PU with CAP.

Apparent viscosity: In these PU paint samples apparent viscosity depends on the polyol content in the sample. Figure 12 showed the same relationship.

C) Discussion on PU paint applied panels

Dry film thickness: The dry film thickness indicates the amount of solids that are retained after the application of spray and this is dependent on the presence of total solid content of the wet mixture to be taken to spraying during the application.

Gloss 60^0 angle: Gloss value depends on the resin solid content in a wet paint sample and surface levelling ability during the drying period. PU paints with CAP, samples have relatively higher gloss values than the other two types but all have exceeded 90. Therefore, all panels have a similar appearance visually and it is very difficult to distinguish to the naked eye.

Water immersion: All PU panels were performed equally and have similar water resistance.

Pencil hardness: All samples exceeded the 2H hardness value. Therefore, all specimens are resistant to scratches and can withstand friction.

Crosshatch test: All panels exceeded 4B states. Therefore, all have good surface adhesion.

Cylindrical mandrel bending test: All panels were properly bent without any crack. Therefore, all panels have similar flexibility.

Impact resistance test: Results of impact resistance are nearly behaved equally. Panels with high film build have more tendency to crack. High-flexibility films show positive results, but brittle films are prone to damage and failure against impacts. Therefore, an impact resistance test can be used to find a high extent of flexible film which has not been detected in the cylindrical mandrel bending test.

Spectrophotometer analysis of color differences: According to table IX, all PU panels furnished by the WBBP have higher CIE DE values and exceeded 2 marks. But PU panels of FBBP were kept lower value as below 1. According to the Figures 17,18 &19 when the CIE DE increases the colour shifts to a more yellow side. Visually also this phenomenon can easily be identified.

CONCLUSIONS

1. PU surface coatings with WBBP were more or less equally performed relative to PU surface coatings with FBBP except colour.

2. When comparing the most critical factors of paint, such as adhesion, flexibility, hardness, resistance to water, and gloss (all rated higher than 90.0), all PU paints perform equally well. Therefore, in terms of properties, PU paint coatings made with waste coconut oil can effectively replace fresh coconut oil and CAP in PU formulations.

3. The CIE DE value is higher in PU paints produced by WBBP, while it is significantly lower in PU paints from FBBP. As a result, WBBP cannot be used for very intense white and light colors. However, FBBP can be utilized for whites and light colors to some extent.

4. During the synthesis of WBBP, the condensation reaction produced relatively low amounts of reaction water, indicating that the degree of polymerization is lower compared to FBBP. As a result, FBBP has a higher molecular weight than WBBP. This difference also demonstrates that FBBP exhibits a higher viscosity than WBBP.

5. In the FTIR spectra of coconut oil, a distinct peak at 2678 cm^{-1} corresponds to the cis distribution of $-C=C$ bonds. This peak gradually disappears, indicating the presence of cis-distributed double bonds exclusively in the unsaturated fatty acids of coconut oil monoglycerides. During the synthesis of BBP, these double bonds are utilized in the formation of other bonds, primarily in the crosslinking between polymer chains. However, this peak is absent in waste coconut oil, suggesting that the domestic cooking process has destroyed the cis –C=C– areas. As a result, Waste BBP (WBBP) has a simpler structure than Fresh BBP (FBBP) and exhibits lower viscosity.

6. PU paints that use WBBP exhibit the shortest touch and hard drying times. This suggests that WBBP contains fewer active hydroxyl (OH) groups available to form urethane linkages compared to FBBP, which results in a quicker curing process. Additionally, during the condensation phase, WBBP shows a relatively high acid value, indicating that certain acids hindered the progress of condensation by affecting the OH groups. Consequently, the domestic cooking process may have damaged some of the active functional groups present in the coconut

oil.

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