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The evolution of palm oil acrylates within 20 years in Nuclear Malaysia

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Introduction

Most of polymeric products available in the market are mainly derived from petroleum feedstock which is non-renewable resources. Due to continuous depletion of fossil oils, the price of this oil was fluctuated and is expected to increase in the new future. On the other hand, the increasing awareness of the consumers on health and environmental issues have changed their preference towards bio-based products or so called 'green' products. Vegetable oils are one of the most important classes of renewable resource. There are various types of vegetable oils that have been utilized or have potential to be utilized in energy and polymer applications such as soybean oil, castor oil, rapeseed oil, rubberseed oil, palm oil, canola oil and jatropha oil. Since Malaysia is one of the world's main producer and exporter of palm oil, it is natural that we should be in the forefront of the research of widening the use of palm oil in oleochemicals industry, which are currently increase in popularity. The presence of unsaturation in the fatty acids of vegetable oils such as palm oil (~ 42%), technically paves the way for the production of acrylated resins. In Malaysia, the production of crude palm oil has increased by 2.30% to 19,216,459 tonnes in 2013 compared to 18,785,030 tonnes in 2012.

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Composition of the Palm Oil – Fatty acid

Asid lemak	Bilangan karbon, C	Bilangan ikatan C=C	Kandungan, %
Asid laurik (C12:0)	12	0	0.1-1.0
Asid miristik (C14:0)	14	0	0.5 - 0.6
Asid palmitik (C16:0)	16	0	32.2 – 45.0
Asid palmitoleik (C16:1)	16	1	0.1- 0.3
Asid stearik (C18:0)	18	0	2.0 – 7.0
Asid oleik (C18:1)	18	1	38.0 – 40.8
Asid linoleik (C18:2)	18	2	5.0 – 11.0
Asid linolenik (C18:3)	18	3	0.0 – 0.6
Asid arakidik (C20:0)	20	0	0.2-0.7

➢ Oxirane oxygen content test (OOC, %) EPOP: **2-3%**
➢ Unsaturated C=C, **42%**

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Strength of the acrylated palm oil

- Renewable natural resources
- Biodegradable
- Environmental friendly
- Non Toxic – Primary skin irritation index (PSI = 2.92), categorized as non irritant
- Competitive price of RM 15/Kg vs petrochemical resin > RM 40/Kg
- Glossiness on wood at 60s: 86-91

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Malaysian market for the acrylated palm oil*

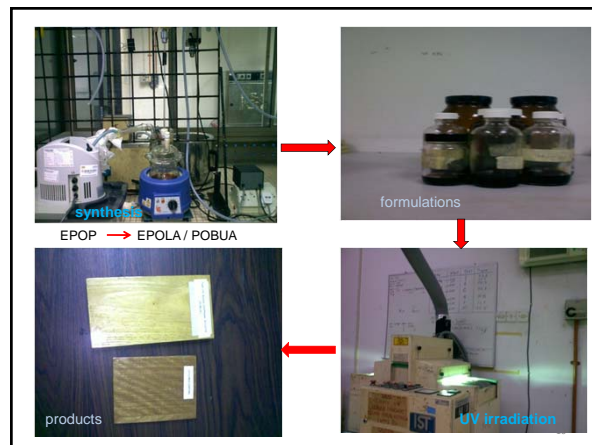
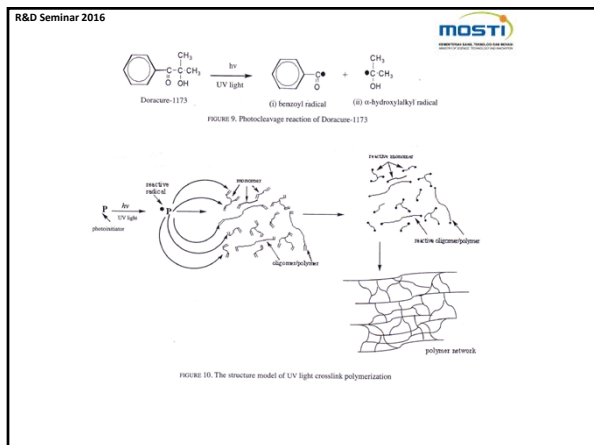
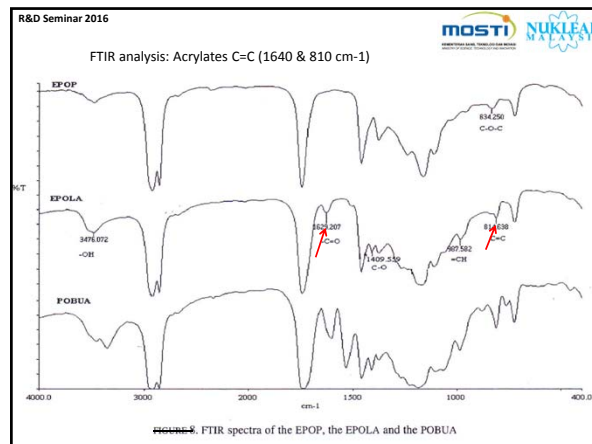
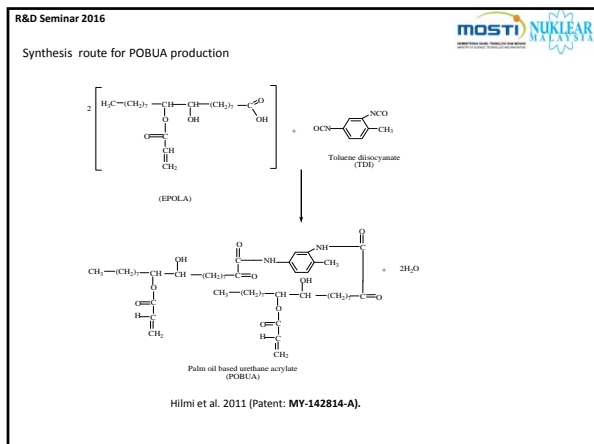
Ref. *Khairul Zaman, M.D. et al, 2011. UV overprint varnish (OPV): Application of epoxidised palm oil acrylate (EPOLA), Proceedings of RadTech Asia 2011, 20-23 June 2011, Yokohama Japan

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Synthesis route for EPOLA production

Synthesized route to produced epoxidised palm oil acrylates /methacrylate*
*Hussin et al.1990; Hilmi et al. 1991



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The Performances of Formulated EPOLA With Various Oligomers and Monomers Cured Using Electron Beam (EB).

Formulations	EPF 1	EPF 2	EPF 3	EPF 4	EPF 5	EPF 6	EPF 7	EPF 8	EPF 9	EPF 10	EPF 11	EPF 12
EPOLA (A.N. 22.42)	30	30	30	30	30	30	30	30	30	30	30	30
Polyester acrylate (pb. 830)	30	30	20	20	30	30	20	20	30	30	20	20
Epoxy acrylate (pb. 625)	30	30	30	30	30	30	30	30	30	30	30	30
Urethane acry (pb. 275)	30	25	35	40	30	25	35	40	30	25	35	40
TPGDA	30	10	10	10	10	10	10	10	10	10	10	10
TMPTA	30	10	10	10	10	10	10	10	10	10	10	10
HEA	30	5	5	5	5	5	5	5	5	5	5	5
Dose (kGy)	30	30	30	30	30	30	30	30	30	30	30	30
Viscosity (cp at 25°C)	138.6	124.0	87.2	94.0	253.8	232.2	123.0	137.5	535.0	477.0	211.5	232.0
Brookfield Hardness (H)	20.04	22.38	22.10	21.07	21.91	21.72	21.44	22.19	12.79	21.67	15.71	14.62
EP Content (%)	89.93	89.27	90.30	90.28	89.20	88.47	89.20	89.43	91.36	91.47	91.47	91.41
Adhesion on wood (%)	100	100	100	100	100	100	100	100	100	100	100	100
Adhesion on wood (at 60°C)	86.0	87.4	84.2	88.7	81.6	86.9	85.1	86.7	86.8	87.3	86.8	86.8
Abrasion Resistance (g weight loss)	0.0296	0.0138	0.0398	0.0145	0.0228	0.0274	0.0224	0.0232	0.0264	n.a	0.0211	0.0284
Tg (°C)	-43	-40	-62	-33	-62	-47	-56	-69	-37	-59	-59	-31
Tensile Strength												
Young's Modulus (MPa)	253.5	231.7	273.1	307.0	n.a	459.6	n.a	495.8	848.3	478.3	367.8	219.1
Stress at break (MPa)	7.49	6.70	6.58	7.96	n.a	6.79	n.a	10.91	4.62	5.36	6.60	6.04
Elongation at break (%)	3.11	4.15	4.77	3.90	n.a	2.38	n.a	3.49	6.62	8.90	6.95	6.05

Abrasion Resistance : g weight loss after a 500 cycles scrubbing with a 500 g load abrasive wheels.
 Tensile Strength : Sample Gauge length = 60 mm, Sample Width = 5 mm, Cross head speed = 10 mm/min
 n.a : Data not available

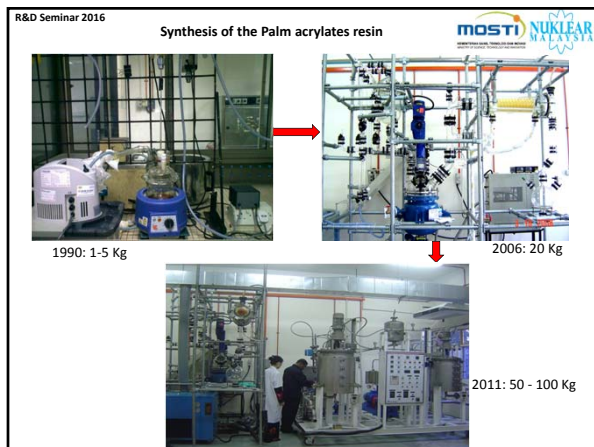
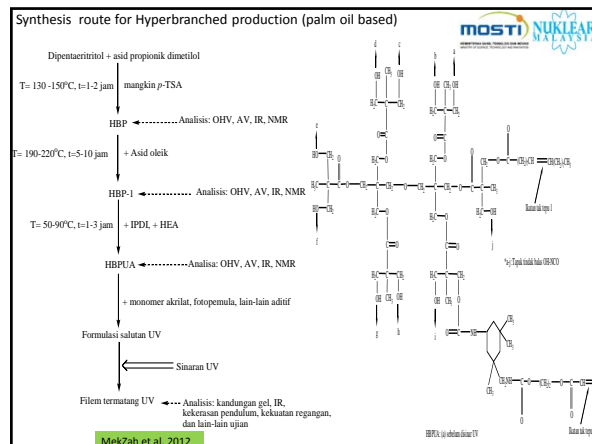
The Physical Properties of POBUA Based Coatings Formulation Cured with 300 KeV Curetron at 30 kGy

	POBUA-1			POBUA-2			POBUA-3			POBUA-4			POBUA-5		
M _w (x 10 ³)	8.998			9.389			9.780			8.422			10.553		
Gel Content (%)	a	b	c	a	b	c	a	b	c	a	b	c	a	b	c
	92.27	92.27	92.27	94.71	94.71	92.45	93.0	92.75	93.04	95.16	94.95	95.14	92.2	94.71	92.25
	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
P.Hardness (%)	35.19	31.07	22.77	37.20	36.16	23.14	37.9	26.04	24.07	28.49	27.68	23.88	30.7	27.32	25.00
Abrasion Resistance (% weight loss)	2.18	0.93	2.27	2.04	1.12	2.47	1.47	1.23	1.47	1.67	1.15	1.62	1.56	1.10	1.10
	Tensile:														
	Stress(MPa)	12.33	13.64	4.55	10.00	14.71	3.64	9.30	11.74	7.80	8.00	10.91	3.33	14.5	17.88
Elongation (%)	6.83	8.83	6.83	8.08	12.39	6.04	6.75	11.47	12.39	7.42	12.08	4.97	13.00	13.00	12.08
60 °C Gloss on Wood Substrate(%)	89.9	90.2	90.3	90.4	90.0	90.1	88.6	91.0	88.5	88.0	90.1	89.2	90.3	89.0	88.9
Adhesion on Wood(%)	96	100		100	92		96	100		100	96		100	100	

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Curing/Coatings Performances of Palm Oil Based Resin (POBUA) vs Petroleum Based

	Petroleum Acrylates	Palm Oil Acrylates
Rate of UV cure (10-25m/min conveyor speed, 7.5 A current)	Varies (structural dependence)	Varies (structural dependence) i.e., 1-5 # passes
Hardness (coatings)	Standard (40-60% Pendulum hardness)	Less/softer (20-40% Pendulum hardness)
Tensile Strength	Standard	Less/inferior
Adhesion (On Paper/PET/ wood substrates)	Standard	Superior (80-100%)
Molecular weight (MW)	Variable/ Adjustable (could be very high, up to 10 ⁶)	Variable/ Adjustable (2,000-20,000)
Glossiness (coatings)	Standard	Standard (60-90° at 60 degree)
Environmental impact	Maximum	Less : -Biodegradable -Environmental friendly - less volatile organic compound
Price	Expensive	Cheaper
Materials resources	Non-renewable	renewable



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Achievements : INNOVATION

No.	Innovation - Medal & event (2003-2015)
1	Gold Medal, Rekacipta & Inovasi ANM 2003 (10-11 Jun 2003) : Sintesis Palm Oil Based Urethane Acrylate (POBUA) & Penggunaannya dalam Coatings, Adhesive & Printing inks.
2	Silver Medal, Expo S&T Kebangsaan 2003 (7-9 August 2003), Kuala Lumpur: Sintesis Palm Oil Based Urethane Acrylate (POBUA) & Penggunaannya dalam Coatings, Adhesive & Printing inks
3	Anugerah Inovasi Perkhidmatan Awam Tahun 2004 (AIPA 2004) : Sintesis Palm Oil Based Urethane Acrylate (POBUA) & Penggunaannya dalam Coatings, Adhesive & Printing inks.
4	Silver Medal, Rekacipta & Inovasi ANM 2004 (22-23 Jun 2004): Penghasilan Adhesif Dari Resin Berasaskan Minyak Sawit".
5	Bronze Medal, Seoul International Invention Fair 2004 : Sintesis Palm Oil Based Urethane Acrylate (POBUA) & Penggunaannya dalam Coatings, Adhesive & Printing inks
6	SILVER MEDAL ITEX 2007 (18-20 May 2007, Kuala Lumpur): New Prospective Palm Oil Based PU/Clay Nanocomposites For Fire Retardant to Complying Sustainable Development
7	BRONZE MEDAL INOVASI NUKLEAR MALAYSIA 2009 : "Palm Oil Based Polyol For the Production of High Functionality Urethane Acrylate and Their Applications in Surface Coatings and Adhesives".
8	BRONZE MEDAL MTE 2009 : High Functional Polyol & PU Elastomer Based On Vegetable Oil-Oleic Acid
9	GOLD MEDAL, MTE 2010, KL (4-6 Feb 2010): Production of Coatings, Adhesives, Printing Inks and Overprint Varnishes (OPV) From High Functional Palm Oil Based Acrylated Resins".
10	GOLD MEDAL, Ekspo Inovasi Islam 2010, Inova'10 USIM , 26-28 September: Production of green coatings, adhesives, printing inks and overprint varnishes (opv) from radiation (uv/eb) curable palm oil based resins

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11	PINGAT EMAS & PEMENANG KESELURUHAN REKACIPTA & INOVASI NUKLEAR MALAYSIA 2010 Hari Rekacipta & Inovasi Nuklear Malaysia 2010 , 26-28 Oktober 2010, DTI, Nuklear Malaysia: "W2W-Conversion of Excess Glycerol-The By Product of Biodiesel Production Into Green Oligomers & Plastic Products".
12	GOLD MEDAL, "The 3rd International Invention Fair of The Middle East 2010 (IIFME 2010)", Kuwait : The Production of Green or Eco-Friendly Coatings, Adhesives, Printing Inks and Overprint Varnishes (OPV) From Radiation (UV/EB) Curable Palm Oil Based Resins".
13	BRONZE MEDAL MTE 2011 , KL . "W2W-Conversion of Excess Glycerol-The By Product of Biodiesel Production Into Green Oligomers & Plastic Products".
14	BRONZE MEDAL Inovasi Nuklear Malaysia 2011 : Hyperbranched (HPB) Polyester Polyol From Palm Oil".
15	SILVER MEDAL Inovasi Nuklear Malaysia 2011 : RADIATION INDUCED FORMATION OF INTERNALLY CROSSLINKED STRUCTURE ACRYLATED PALM OIL (APO) NANOPARTICLES USING OIL IN WATER MICROEMULSION SYSTEMS".
16	3rd prize Best Poster Award, IMTCE 2014 , 14-15 Mei 2014, Kuala Lumpur: The production of Hyperbranched Curable Palm oil Oleic acid
17	Silver Award, (I-INOVA) 2015 , 24-25 th Oct 2015, USIM, Negeri Sembilan: Palm Oil based Eco-Friendly Printing Inks Materials



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Achievements: Patent

No	IP (2003 -2013)
1	MY-142814-A (2011): Process for the production of palm oil based containing products (polyols/diols) for use in making polyurethane materials
2	PI 20031627: Synthesis and production of palm oil based urethane (POBUA) for use in UV/EB curing of coatings, adhesives and printing ink.
3	PI 20040082: Radiation curable pressure sensitive adhesives (PSA) from palm oil based resins/oligomers and manufacturing method there of
4	PI 2013701558: A composition susceptible to irradiation for use as a compounder carrier.

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INVENTION synopsis

Patent number : MY 142814-A
Filing Number : PI 20031627

METHOD FOR MANUFACTURING PALM OIL BASED HYDROXYL CONTAINING PRODUCTS FOR USE IN MAKING POLYURETHANE MATERIALS

The present invention provides a method for manufacturing palm oil based polyols or diols having moderate molecular weight of between 500 and 1000 and a desired hydroxyl content of between 2 and 4 that make it suitable for use as starting materials making polyurethane based materials that include adhesives and coatings. The palm oil based polyols or diols are made by way of acylation or methacrylation using acrylic acids or methacrylic acids or any other suitable product of acrylic acid with the addition of stabilizer and catalyst under appropriate conditions. Hydroxylation of palm olein based olein acid by way of direct partial modification is carried out with the addition of catalyst and stabilizer. Hydroxylation of palm olein by way of reacting double bond in said palm olein structure using hydrogen peroxide is achieved with the addition of catalyst and hydroxylation of palm olein by way of oxidation using a hydrogen peroxide catalytic acid system.

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INVENTION synopsis

Filing Number : PI 20030227

SYNTHESIS AND PRODUCTION OF PALM OIL BASED URETHANE (POBUA) FOR USE IN UV/EB CURING OF COATINGS, ADHESIVES AND PRINTING INK.

New radiation curable palm oil based urethane acrylate or methacrylate are prepared by reacting epoxidised palm oil acrylate or methacrylate or any other functional containing palm oil based products developed by Nuclear Malaysia, all of them refer to as Nuclear Malaysia product or a mixture thereof with a diisocyanate and a hydroxyl group containing acrylate or methacrylate or polyacrylate or polyurethane.

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Products: Inks, Paper Coatings, Wood Coatings

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INVENTION synopsis

Filing Number : PI 20030227

RADIATION CURABLE PRESSURE SENSITIVE ADHESIVES (PSA) FROM PALM OIL BASED RESINS/OLIGOMERS AND MANUFACTURING METHOD THEREOF

A method of producing a non-permanent type radiation curable pressure sensitive adhesives (PSA) from palm oil based olein acids, and more particularly palm oil based olein acids olefin or alginates is provided, wherein, the palm oil based mon/oligomer is reacted with acrylate or methacrylate monomers in the presence of stabilizer resin and additive at a temperature range between 30°C, 40°C, and undergoes mixing and/or pre-irradiation mixing, and irradiated in suitable prior to using with electron beam or UV based irradiation.

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Pressure sensitive adhesives (PSA)

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Publications: Journal

No	Journal (1991- 2015)
1	Mohd. Hilmi, M., Hussin Mohd. Nor, Hamirun Kifli and Masni Abdul Rahman, (1991), The Use of Epoxidised Palm Oil Products (EPOP) For The Synthesis of Radiation Curable Resins-11. Ultraviolet (UV) Curing of Epoxidised RBD Palm Olein Acrylate (EPOLA), <i>Nucl. Sci. J. Mal.</i> 9(2): 95-102
2	Mohd Hilmi Mahmood, Dahlan Hj Mohd., Abd Ghani Harun, Ahmad Shakeri Mat Seman and Aizool Abd Kadir (1992), UV Curing of Epoxidised Palm Oil Acrylate (EPOLA) and Liquid Epoxidised Natural Rubber Acrylate (LENRA) On Rubber Wood Substrates-A Preliminary Study, <i>Nuclear Science Journal of Malaysia</i> , Vol 10 No 182, December 1992, pages 1-5
3	Mohd Hilmi Mahmood, M. N. Munawar Hussin, Mat Rohani Omar, Roslan Ismail, Salmiah Ahmad and Hazimah Abu Hassan (1993), Studies on UV curing of palm oil acrylate (EPOLA) coatings. The Effect of EPOLA viscosity, oligomer and monomer or crosslinker contents and photoinitiator combinations on curing rates and cured film properties, <i>Nuclear Science Journal of Malaysia</i> , Vol 11, No 3, June 1993, pages 1-10
4	Mohd Hilmi Mahmood, Hajar Mohd Nor, Khairul Haidi Abd Raof, Roslan Ismail, Mohd Rosli Mohd Razbi, Saiful Mohd Shariff and Zahid Abdullah (1995), Development of pressure sensitive adhesives (PSA) from palm oil acrylated resins by irradiation (UV/EB), <i>Nuclear Science Journal of Malaysia</i> , Vol 13, No 2, Dec. 1995, pages 61-68
5	Mohd. Hilmi Mahmood, Zahid Abdullah, Yasuo Sakurai & Khairul Zaman, Effects of Monomers On The Properties of Radiation Curable PSA-A prepolymer Method, <i>Radiation Physics & Chemistry Journal</i> , Elsevier Sci. Publisher, Pergamon Press U.K., 20 (1-2), Jan. 2001, pp 129-137.
6	Mohd. Hilmi Mahmood, Shahrol Najmin Baharom, Rosley Che Ismail, & Khairul Zaman Hj. Mohd. Dahlan, Effects of structures and molecular weight on the properties of EPOLA based radiation curable pressure sensitive adhesives (PSA), <i>Nuclear Science Journal of Malaysia (NSJM)</i> , Vol. 19, No. 182, June & December 2001, pages 70-78.
7	W. D. Wan Rosli, RN Kumar, S. Mek Zah and M. Mohd Hilmi, UV radiation curing of epoxidised palm oil-cycloaliphatic diisopropyl ether system induced by cationic photoinitiators for surface coatings, <i>European Polymer Journal</i> , Vol 39, Issue 3, March 2003, pages 593-600.
8	M. Hilmi M, Shahrol Najmin B, Mek Zah S., Khairul Zaman H.M.D. & Rosley C.I., Effect of Structure and Molecular Weight On Properties of Pressure Sensitive Adhesives (PSA) Formulated From Palm Oil Based Urethane Acrylate (POBUA), <i>Journal of Nuclear And Related Technologies</i> , Vol 1, No. 2, Dec 2004, pages 32-37.


No.	Journal
9	T. Rihayat, Saari M, M Hilmi Mahmood, Wan Md Zin Wan Yunus, Suraya A R & K Zaman M Dahlan, Thermoplastic PU Elastomers Based On Palm Oil Polyol, <i>Journal of Applied Technology</i> , Vol 3 No 1, Mei 2005 (71-76).
10	T. Rihayat, S.B. Mustapha, M.Hilmi Mahmood, W. MD Zin W. Y., S.A. Rasid and Khairul Zaman H.M.D., Study on Formulation of PU/Clay Nanocomposites Based On Palm Oil Polyol, <i>Journal of Science & Technology</i> , Vol. 3, No. 6 December 2005, pages 1-4.
11	T. Rihayat, S.B. Mustapha, A. R. Suraya, M.Hilmi Mahmood, Khairul Zaman H.M.D, W. MD Zin W. Y., and S. M. Sapuan., Synthesis and Thermal characterization of PU/Clay Nanocomposites Based On Palm Oil Polyol, <i>Journal of Polymer-Plastics Technology and Engineering</i> , Vol. 25, No. 12 December 2006, pages 1323-1326
12	T. Rihayat, SB Mustapha, M. Hilmi Mahmood, WMZ Wan Yunus, AR Suraya, KZHM Dahlan and SM Sapuan, Mechanical Characterization of Palm Oil Based PU/Clay Nanocomposites, <i>Polymers & Polymers Composites</i> , Vol 15, No 8, 2007, pgs 597-602.
13	Yaakob, Z., Min Min A., Mohd Hilmi M., Khairul Zaman Hj M.D and kamaruddin S.K.K., Effect of Compatibilization Of Polymer On the properties of Polyurethane-Palm Fibre Composites, <i>Journal of Polymer Engineering</i> , Vol 29, Nos. 8-9, 2009, pages 503-520.
14	M.F. Juhaida a,1, M.T. Paridah b,* , M. Mohd. Hilmi c, Z. Sarani d, H. Jalaluddin b, A.R. Mohamad Zaki c, Liquefaction of kenaf (Hibiscus cannabinus L.) core for wood laminating adhesive, <i>Bioresource Technology</i> 101 (2010), pages 1355-1360.
15	Mek Zah Salleh, Khairiah Badri, Mohd Hilmi Mahmood and Sharim Ahmad, Synthesis of UV Curable Hyperbranched Urethane Acrylate From Palm Oil Oleic Acid, <i>Journal Nuclear and Related Technologies</i> Vol.7, No.2, December 2010.
16	Abdul Amir H. Kadhum,1,a, Mohd Najib Baharu,1,b and Mohd Hilmi Mahmood,2, Elastic Polyesters from Glycerol and Azelaic Acid, <i>Advanced Materials Research</i> , Vols 233-235, May 2011, pages 2571-2575.
17	Mek Zah Salleh, Khairiah Badri and Mohd Hilmi Mahmood. 2013 Synthesis of EB-Curable Hyperbranched Urethane Acrylate From Palm Oil Oleic Acid. <i>Int. J. Materials Engineering Innovation</i> , (2013), Vol.4, No.1:65-78.
18	Mek Zah Salleh, Khairiah Badri, Sahrim Hj Ahmad, Mohd Hilmi Mahmood, Rida Tajau, Nik Ghazali Salleh & Mohamed Lokman Latif. 2013. Properties of Radiation Curable Hyperbranched Polyurethane Acrylate from Olein Acid of Palm Oil. <i>J. Nuclear Science & Techniques</i> , 2013, 24(5):S010303
19	Tajau, R., Wan Yunus, W. M. Z., Mohd Dahlan, K. Z., Mahmood, M. H., Hashim, K., Salleh, M., Ismail, M., Che Ismail, R. Radiation-Induced Formation of Acrylated Palm Oil Nanoparticle Using Pluronic F-127 Microemulsion System. <i>Pertanika J. Sci. & Technol.</i> 21 (1): 135- 142 (2013)

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No.	Journal
20	Rida Tajau, Mohd Hilmi Mahmood, Mek Zah Salleh, Khairul Zaman Mohd Dahlan, Rosley Che Ismail, Sharifa Muhammad Faisal & Sheikh Mohd Zaki Sheikh Abdul. Production of UV-Curable Palm Oil Resin/Oligomers Using Laboratory Scale and Pilot Scale Systems. <i>Sains Malaysiana</i> 42(4)(2013): 459-467
21	Mek Zah Salleh, Khairiah Badri, Rida Tajau and Nik Ghazali Salleh. 2014. The production of green polymer hyperbranched curable palm oil oleic acid. <i>J. Advanced Materials Research Vol.</i> 1024: 197-200


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Research Grant

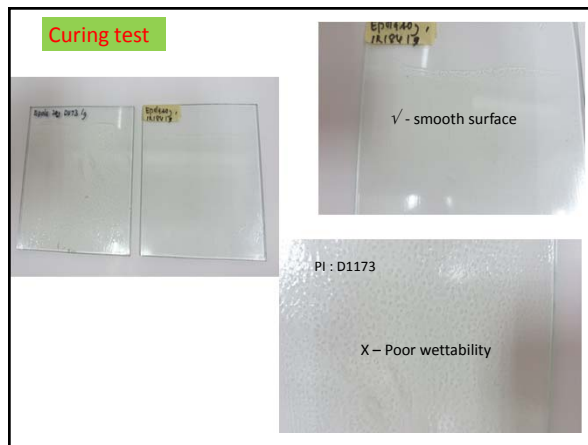
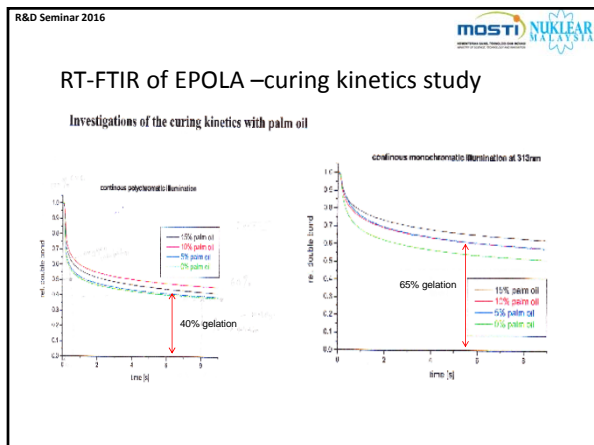
Year	Fund/Grant	Amount (RM)
1995-1997	Project Code: IRPA 03-01-03-005: Radiation Curable Resins from Palm Oil	2,597,964
1996-1997	Research Contract : MINT/UPP/96/05: Radiation Curing of Coating for Fertilizers	30,000
2007-2008	RMC-MINT-R&D-05-050: High Functionality Polyol/PU	20,000
2009-2011	Trust Fund ANM: Pilot Plant for Synthesis Epoxy Acrylate (EPOLA) and Formulations of Overprint Varnishes (OPV)	500,000
2011-2013	Scifund MOSTI: 03-03-01-SF0052: Development of Nano Particles of Epoxidised RBD palm Oil Acrylate (EPOLA) As Drug Carrier In Delivery System Using Ionizing Radiation Technique	173,000
2011-2013	Scifund MOSTI: 03-03-01-SF0164: Development of Hyperbranched Curable Resin from Palm Oil	233,000
2012-2013	Scifund MOSTI: 03-03-01-SF0162: UV Spot OPV Based on Acrylated Palm Oil Resin (EPOLA)	40,000
2015-2017	Dana Peruntukan Khas MOSTI (PKA05140002): Development of Radiation Curable Overprint Varnishes (OPV) Based on palm Oil Acrylate (EPOLA) Resin	316,000
1995-2016	Total	3,889,964

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Limitation used of the acrylated Veg. Oil –Palm acrylates:

- Poor wettability – selected photoinitiators, monomers.
- low crosslinking – 1-2 functional groups & 40-65% gelation



R&D Seminar 2016



Conclusion

- Suitable application of the Palm acrylates (EPOLA/POBUA) as an additives to improve glossiness in the coating film (Printing inks, OPV etc)
- Still searching the company/Partner to transfer the product/technology

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