

**The Synthesis of Polyol from Rice Bran Oil
(RBO) through Epoxidation and
Hydroxylation Reactions**

by

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Declaration

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Summary

Polyurethanes are valuable polymers with a wide variety of applications. They are normally produced from polyol feedstocks derived from petroleum. As petroleum is a non-renewable resource, an alternative source of feedstock is sought. A potential source is rice bran oil. However, far too little attention has been paid to the utilization of rice bran oil as a potential raw material to produce polyol as it contains unsaturated fatty acids that can be converted to polyol and is the by product of rice milling process and available at very low cost.

There are two sequential processes to produce polyol from rice bran oil, namely the epoxidation and hydroxylation reactions. In this work, the optimal conditions in the epoxidation reaction were investigated using acetic acid and formic acid as oxygen carriers in terms of reaction time and temperature. Furthermore, the reaction kinetics were also determined using formic acid as an oxygen carrier in the epoxidation step. Finally, the influence of reaction time and temperature in the hydroxylation step were also investigated in this study.

In order to determine the optimal condition, the epoxidation reaction was performed in a three neck flask with the use of acetic and formic acid as oxygen carriers. Result shows that the conversion of iodine value increased with reaction time and temperature when acetic acid was used as an oxygen carrier (peroxyacetic acid). Interestingly, the oxirane content increased with reaction time and temperature then declined after having achieved the optimal point. The optimal condition was achieved at a reaction time of 4.3 h and a temperature of 63.8°C by performing response surface methodology.

The conversion of iodine value also displayed similar behaviour during the epoxidation reaction when formic acid was used as an oxygen carrier (peroxyformic acid), namely the conversion increased with reaction time and temperature. The measured rate constants were $0.172\text{h}^{-1}(40^{\circ}\text{C})$, $0.304\text{h}^{-1}(50^{\circ}\text{C})$, $0.374\text{h}^{-1}(60^{\circ}\text{C})$, $0.425\text{h}^{-1}(70^{\circ}\text{C})$ and $0.492\text{h}^{-1}(80^{\circ}\text{C})$. The activation energy was 22.6 kJ/mol and the epoxidation reaction was pseudo-first order with respect to the concentration of

double bonds in the oil. Interestingly, peroxyformic acid shows improved performance as indicated by higher content of maximal oxirane content 3.26% compared to peroxyacetic acid which is only 2.62%. The optimal condition with the use of formic acid as an oxygen carrier was obtained at reaction time of 4 h and temperature of 60°C.

In the hydroxylation step, results indicate that the hydroxyl value of polyol was a quadratic function of reaction time and temperature and the optimal condition was achieved at a reaction time of 125.5 min and temperature of 49°C, with maximal hydroxyl value 161.5 mg KOH/g oil by performing response surface methodology. The viscosity of polyol increased with reaction time and temperature and resulted in polyol with viscosity in the range 29.9 – 95.3 cP. Temperature was found to have the most significant effect on the viscosity of polyol.

The results of this study confirm the potential of rice bran oil as a feedstock for synthesis of polyol and show that the optimal conditions in the epoxidation and hydroxylation reactions are a key control variable to obtain a high quality of polyol.

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I would like to dedicate this thesis to my wife, Nanik Hasanah and my son, Nawfal Adiva Purwanto.

I hope this thesis would provide a great contribution to the community and satisfy with the expectations of the related people.

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Nomenclature

E	activation energy (kJ/mol)
k	reaction rate constant (h^{-1})
m	mass (g)
n	reaction orders
n	moles number (mol)
N	normality (N)
R	gas constant (J/mol.K)
R^2	correlation coefficient
SA	saponification value after acetylation (mg KOH/g oil)
SB	saponification value before acetylation (mg KOH/g oil)
t	reaction time (h, for epoxidation reaction)
t	reaction time (min, for hydroxylation reaction)
T	temperature (K)
V	volume (mL)
x	real value of independent variables
X	coded value of independent variables
X	reaction conversion
Y	response variables

Greek

Δx	step change value
ρ	density (g/mL)
τ	shear stress (Pa)
$\dot{\gamma}$	shear rate (s^{-1})
μ	viscosity (cP)

Abbreviations

ANOVA	analysis of variance
DB	double bonds
EO	epoxidized oil
FA	formic acid

FFA	free fatty acids
HV	hydroxyl value
IV	iodine value
MMT	million metric tons
MW	molecular weight
PFA	peroxyformic acid
PL	phospholipids
PPO	polypropylene oxide
RBO	rice bran oil
RSM	response surface methodology
US	united states