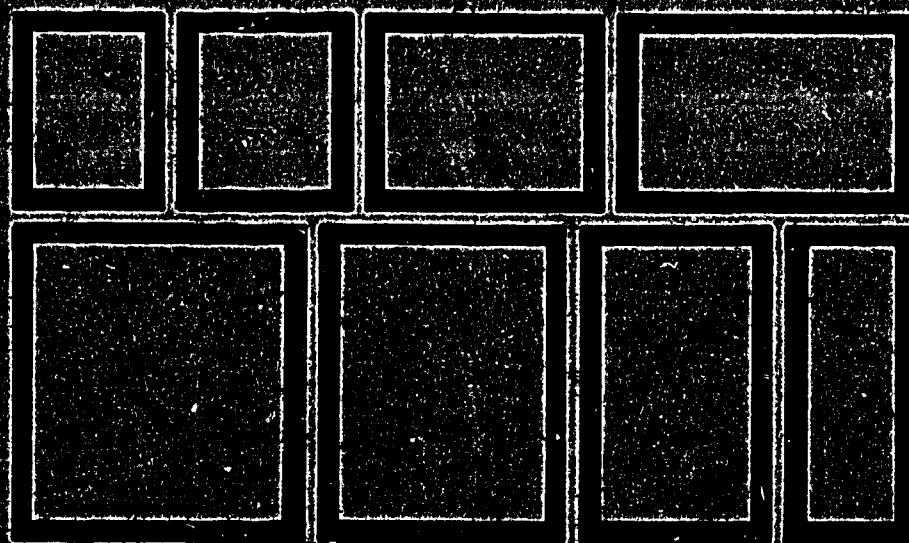


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SYNTHESIS OF EPOXIDISED SOYA BEAN OIL ACRYLATE



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SYNTHESIS OF EPOXIDISED SOYA BEAN OIL ACRYLATE

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Abstract

An epoxy acrylate was synthesised from Asahi's epoxy resin AER 331 which is an epoxidised soya bean oil (ESBO). Triethylamine (TEA) and Hydroquinone (HQ) were used as catalyst and inhibitor respectively. Observations of the experiment are described.

Introduction

ESBO can be obtained via epoxidation of soya bean oil using peracetic acid or hydrogen peroxide¹. The epoxidised oil, typically, contains between 3 to 4 epoxy groups per molecule. Their epoxy groups can be reacted with acrylic or methacrylic acids to produce epoxidised soya bean oil acrylate (ESBOA). The epoxy acrylate molecules contain unsaturation and hydroxyl groups and hence the molecule can be polymerised through the double bond, as well as, adhesion and pigment wetting properties are improved by the presence of the hydroxyl groups².

The properties of epoxidized oils acrylates are shown in table 1:-

Table 1: Properties of the Acrylated Oils³

Advantages	Disadvantages
Excellent pigment wetting	Slow cure
Low cost	Soft film
Good adhesion	
Low skin irritancy	

The disadvantages obviously limit their use. The low skin irritancy and good pigment wetting properties make them attractive for use in ink formulations. Other applications include paper and board varnishes, overprint lacquers and wood finishes³.

Experimental

Materials

Epoxy resin used in this work was epoxy resin AER 331 supplied by Asahi Company. Its properties are shown in table 2.

Table 2: Properties of Epoxy Resin AER 331

! Trade Name	! AER 331	!
! Equivalent Weight, g	! 180-200	!
! Viscosity, cps/25°C	! 12000-16000	!
! Colour gardner	! 2	!
! Density, 25/25	! 1.16	!
! Flash Point, °C	! 292	!
! Temp. °C/1~2ps	! 70	!

Acrylic acid, 99% (merck) was purified by reduced pressure distillation technique prior to use. Other chemicals (merck) were hydroquinone and triethylamine which were used as inhibitor and catalyst respectively.

Apparatus

The apparatus used in this work consists of a vessel (reaction flask) equipped with a mechanical agitator, a reflux condenser, a nitrogen gas inlet, a dropping funnel and a bent thermometer. The apparatus set up is shown in figure 1.

Acrylation of ESBO

The ingredients employed in this experiment is shown in table 3.

Table 3: Ingredients for Acrylation

! Epoxy resin AER 331 (Asahi) -	47.70g	!
! Acrylic acid	- 16.77g	!
! Hydroquinone	- 0.32g (0.5%)	!
! Triethylamine	- 0.65g (1.0%)	!

The process of acrylation is:-

! Charge the resin, hydroquinone, triethylamine	!
!	!
! Start nitrogen purge and agitate. Heat to	!
! 80°C and then charge the acid	!
!	!
! Increase and maintain temperature at 100°C	!
! until the acid value is < 5mg KOH/g	!

Procedure and Observations

47.70g of the Epoxy resin AER 331 having equivalent weight between 180-200g was charged into five necked flask. Then 0.3g of hydroquinone and 0.65g of triethylamine were added. A nitrogen flow was introduced through a gas inlet and maintained until the end of the preparation. The synthesis was started with stirring and heating the mixture to

80°C. The colour change (from colourless to yellow and dark yellow/brown) of the mixture occurred during the heating which was suspectedly due to the formation of the complex between triethylamine and epoxy resin.

When the temperature in the reaction flask reached 80°C, 16.70g of acrylic acid was introduced to the resin via a dropping funnel at a very slow rate. A cloudy atmosphere was observed in the reaction flask due to the evaporation of triethylamine and acrylic acid, but after the reaction proceeded the atmosphere in the flask was clear again. Since the reaction is exothermic, the temperature may be raised above 80°C. In such a case, the heating mantle should be turned off. The colour of the mixture (brown) changed from brown to yellow upon the addition of acrylic acid, due to the formation of epoxy acrylate.

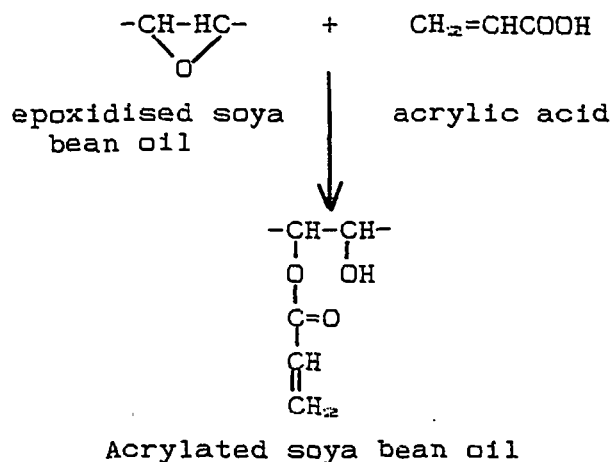
After the addition of acrylic acid was completed, the temperature was increased to 100°C and then maintained until the acid value of the resin is less than 5mg KOH/g sample. It is important to maintain the temperature below 100°C in order to avoid gelation (see the appendix for the procedure of acid value measurement). The stirring rate was increased as the viscosity of mixture increased. In this work, the synthesis was terminated, when the acid value was 1.0 mg KOH/g resin. The product was a very viscous acrylic prepolymer and dark yellow in colour.

Infra-red Measurements

Infra-red spectra of the ESBO (AER 331R, Asahi Japan) and ESBOA were obtained by using Nicolet 60 SX FTIR spectrophotometer.

Discussion

As mentioned earlier, the reaction involved in this synthesis is between epoxy group and acrylic acid which can be summarised in the following equation:-



It is important⁴ to mention here that gelation is the hindrant factor in the synthesis of epoxy acrylates. Two factors which might be contributory to the gelation are the addition rate of acrylic acid and temperature. The addition of acrylic acid must be at a very slow rate. During synthesis, the temperature should not be exceeded above 120°C.

The ESBO and ESBOA IR spectra are shown in figure 2 and 3 respectively. Figure 2 shows a peak at 915cm^{-1} which belongs to the epoxy groups. On the contrary, the peak does not show up in the ESBOA spectrum (figure 3) suggesting that the acrylation reaction between the epoxy group and acrylic acid has taken place. To confirm even further, a peak at 1635cm^{-1} representing acrylic group appears in the spectrum (figure 3).

Acknowledgement

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Appendix

Acid Value Determination

At first, dissolve 1-2 g of resin with 10 ml acetone and then add a few drops of phenol red : bromothymol blue (1:1 in 90% ethanol) indicator. After that titrate with 0.1N KOH which is previously determined its f factor, using 0.1N HCl as titrant and phenolphthalein as indicator.

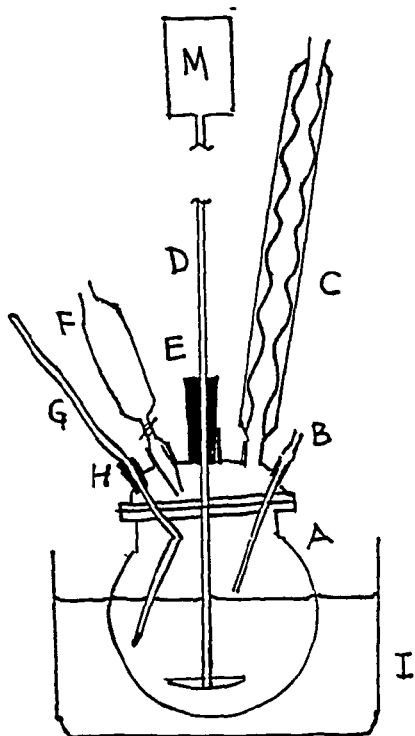
Acid value is the number of milligrams KOH required to neutralize 1 g of resin and calculated by using the following formula.

$$AN = \frac{\text{mg KOH}}{\text{1g of resin}} = \frac{5.61 \times f \times v}{w}$$

where f is the factor

v is volume of KOH solution

w is weight of resin sample



- A. Separable reaction flask
- B. Nitrogen gas inlet
- C. Reflux condenser
- D. Stirring rod
- E. Stirring seal
- F. Dropping funnel
- G. Bent thermometer
- H. Thermometer holder
- I. Oil bath
- M. Mechanical agitator.

FIGURE 1

Figure 2: FTIR Spectrum of ESBO

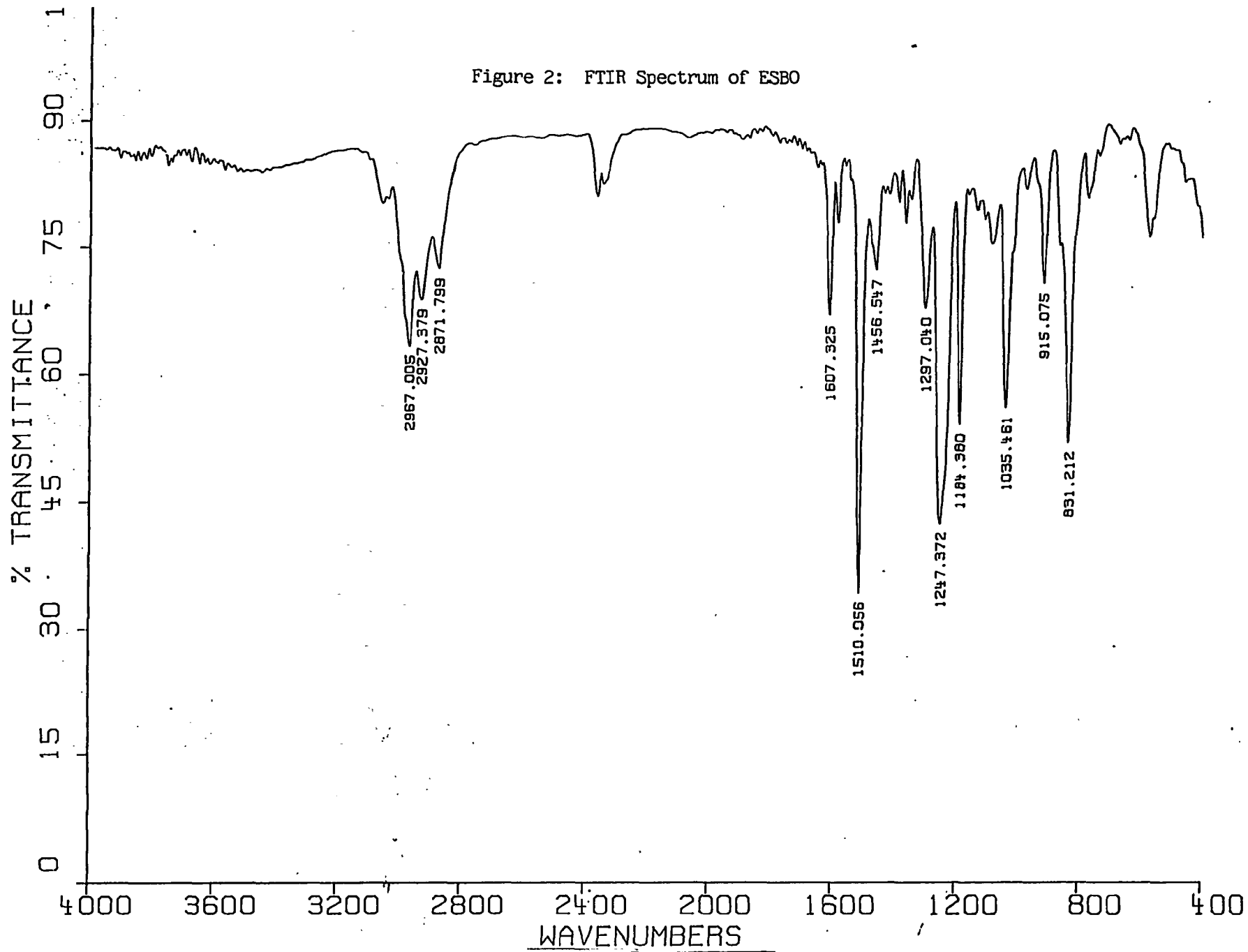


Figure 3: FTIR Spectrum of ESBOA

