RAPID METHOD FOR THE DETERMINATION OF HYDROXYL VALUE IN PALM-BASED POLYOLS

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Hydroxyl value is defined as the number of hydroxyl end-groups per gram of a sample. It is a very important quality parameter, and a typical measure to determine molecular weight and calculate the functionality of polyols. A polyol is an alcohol with more than two reactive hydroxyl groups per molecule. Polyols are the starting material for producing polyurethane. Polyols when reacted with an appropriate isocyanate and a suitable blowing agent will produce polyurethane. The hydroxyl value of a polyol is closely associated to the physical and mechanical properties of the resultant polyurethane foam. Therefore, any change in the hydroxyl value may result in a change in the quality of the polyurethane foam produced. Hydroxyl value is also important in polyol analysis to gauge the extent of a reaction, or the number of hydroxyl groups for further reaction. Hydroxyl value is a critical parameter routinely monitored by polyol manufacturers to ensure the quality of the polyols produced.

For the purpose of on-site quality control in polyols production, a rapid method which does not involve much sample preparation and lengthy steps is desired. The current standard method is the AOCS Standard Method which involves titration that can be extremely time-consuming and is subject to variation from analyst to analyst. Furthermore, this standard chemical method uses toxic chemicals such as pyridine and acetic anhydride which can be detrimental to health. A Near Infra-red (NIR) Spectroscopy method (*Figure 1*) with a partial least square algorithm has been developed using palm-based polyols as the calibration standards. This method allows the measurement of the hydroxyl value in a push button manner. No complicated handling is involved, and the method can be performed easily by any non-technical staff. The method also does not use any hazardous chemicals.

SCOPE

This test method is for the determination of hydroxyl value of palm-based polyols.

PRINCIPLES OF THE METHOD

- Various types of palm-based polyol samples (*Figure* 2) with hydroxyl values ranging from 50 to 250 mg KOH g^{-1} were used as calibration standards.

Figure 1. Near Infra-red (NIR) spectroscopy for the determination of hydroxyl values of palm-based polyols.

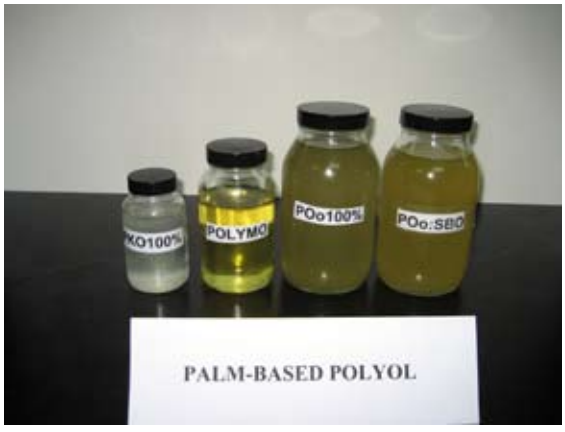

Figure 2. Palm-based polyols.

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- The calibration standards palm-based polyols were homogenized in an oven at 50°C and transferred into NIR glass vials.
- The correlation coefficient (R^2) of the palm-based polyols predicted by the instrument using the partial least square algorithm was established.
- The correlation coefficients of the palm-based polyols calibration standards predicted by the instrument were good, ranging from 0.98-0.99, with good residual errors of mostly below 3.5 for the first hydroxyl overtone region (6500-8100 cm^{-1}) (*Figure 3*) and the full NIR region (4500-10 005 cm^{-1}).
- The calibration plot was then validated with samples of known hydroxyl values, and a validation plot was obtained with correlation coefficients of 0.98-0.99 (*Figure 4*) and good residual errors of below 3.5.
- Once the calibration plot was established and validated, the hydroxyl values of any unknown palm-based polyols can be determined within 2 min.
- The range of hydroxyl value calibration can be expanded if palm-based polyols with lower or higher hydroxyl values are to be monitored.

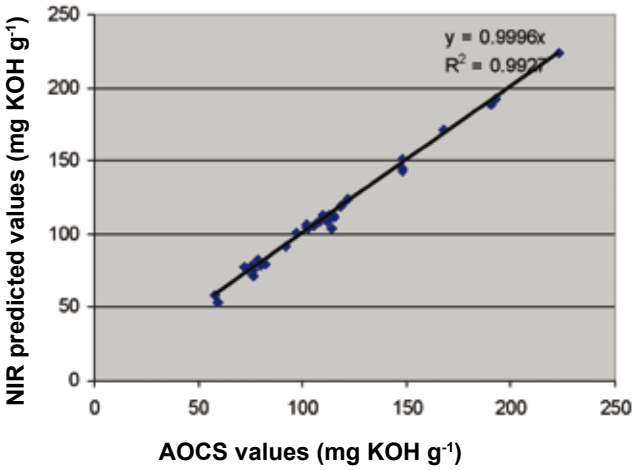

Figure 3. NIR calibration plot for the first hydroxyl overtone region (6500-8100 cm^{-1}) with a correlation coefficient of 0.993.

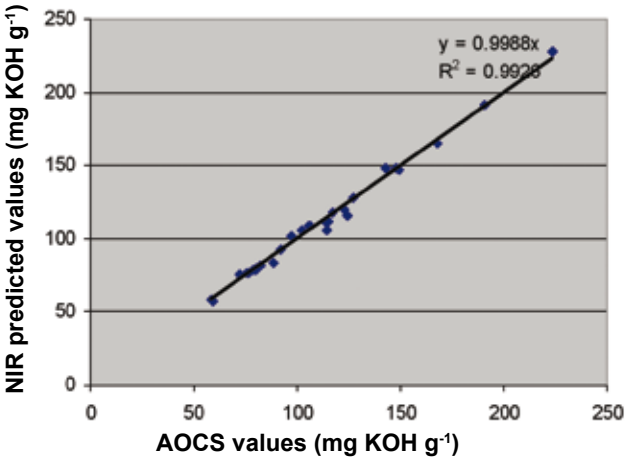

Figure 4. NIR external validation plot for the region (6500-8100 cm^{-1}) with a correlation coefficient of 0.993.

ADVANTAGES OF THE NIR METHOD OVER THE CONVENTIONAL AOCS METHOD

- The method does not involve the use of hazardous chemicals.
- Sample preparation is simple.
- Analysis time is reduced from 2 hr by the AOCS method to about 2 min per sample.
- More samples can be analysed per day compared to the conventional AOCS standard method.
- The method is instrument-based and is 'on-site quality control' friendly.
- It is less costly in the long run because no chemicals, glassware, water bath or fume hood is used.
- Analysis can be carried out by non-technical staff. It is as easy as a 'push-button' operation.

TRANSFER OF METHOD

MPOB may assist any palm-based polyol producer to set up this test method in-house.

REFERENCE

AOCS (2007). Hydroxyl value. *AOCS Official Method*. 5th edition. American Oil Chemist's Society, Urbana, IL, USA.

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