



Photocrosslinkable modified vegetable oil based resin for wood surface coating application

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ABSTRACT

An acrylated epoxidized linseed oil (AELO) was synthesized from epoxidized linseed oil through ring opening of the oxirane group using acrylic acid as ring opening agent. The occurrence of the acrylate group and the ring opening of oxirane group was monitored using FT-IR spectroscopy. The AELO was mixed with three different photoinitiators at two different concentrations. Wood surfaces were coated with the mixtures, subsequently cured under UV light and the resulting surface properties of the coated samples gloss, scratch resistance, solvent resistance, and coating adhesion were characterized. The efficiency of the photoinitiators and the influence of their concentration on the rate and the extent of the curing were studied by curing the AELO mixtures under a monochromatic wavelength of 365 nm and measuring absorption spectra during the cure by real time FT-IR spectroscopy. The decrease of absorption in the measured spectra at 1406 cm^{-1} was used to calculate the conversion of acrylic double bonds with increasing time of UV light exposure to obtain information on the cure kinetics for each photoinitiator and concentration.

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1. Introduction

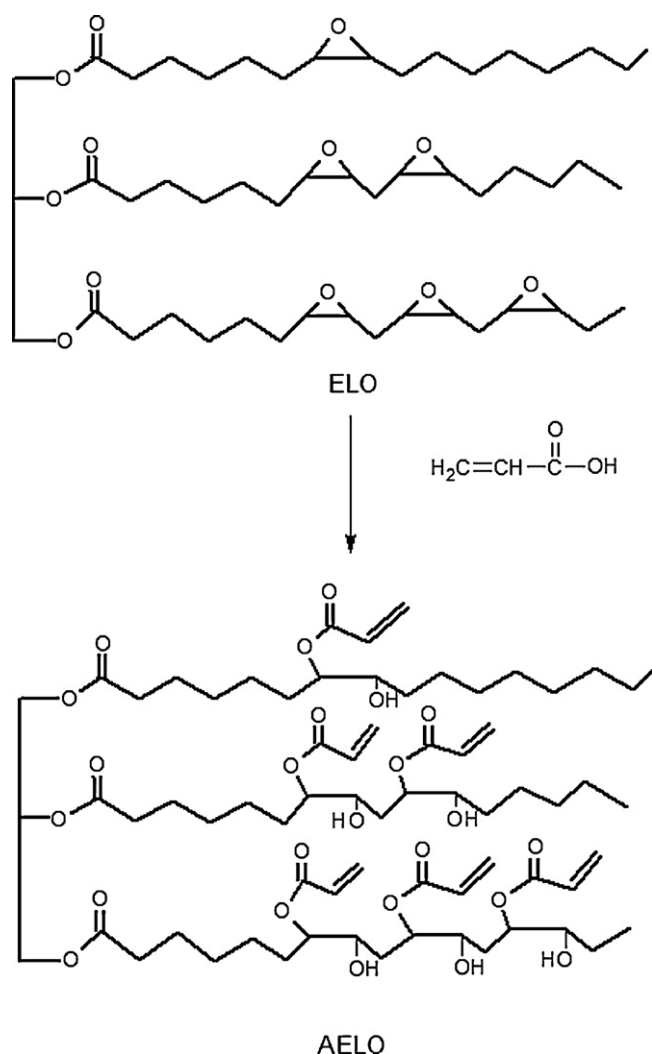
Currently, there is an increasing interest in the use of renewable resources as alternative raw materials for polymeric binder and coating systems which is motivated by two major reasons: firstly, an increasing public awareness of environmental issues and the related demand of “green” products and secondly, an increase in crude oil price due to shortage of fossil resources. The emission of volatile organic compounds (VOC) from industrial products can be reduced or even avoided by the use of renewable resins. Natural fats and oils are among the most promising renewable raw materials used by the polymer industries as a basis for natural-derived binder and coating systems due to their availability, wide range of chemical functionalization and relatively moderate and stable market prices. Since vegetable oils are non-toxic, non-pollutant, and biodegradable they may be used as replacements for petroleum based products used in printing inks, adhesives and coating applications. A wide range of polyfunctional acrylate monomers and

oligomers of vegetable oils have already been prepared from epoxidized soybean, castor, lesquerella, palm und vernonia oil [1–8]. These vegetable oils are based on triglyceride of different fatty acids with varying degrees of unsaturation. Among the triglyceride oils, linseed oil is commercially used as drying oil in paints and protective coatings due to presence of large amount of α -linolenic acid compared to other vegetable oils, which has reactivity towards oxygen in air. The α -linolenic acid content is around 51–55% [9]. The double bonds in the fatty acid chain of the linseed oil are unable to homopolymerize [10], since they show low reactivity. So the double bonds present in the triglyceride chains can be modified to tailor their properties. In the present investigation epoxidized linseed oil was acrylated using acrylic acid and the ring opening of the oxirane ring (Scheme 1) was characterized using FT-IR spectroscopy.

The polymerization and crosslinking of reactive substrates by UV curing was introduced in the 1960s in the wood and furniture industries. This UV curing technology offers many advantages like solvent-free polymerization, low energy consumption, and rapid cure speeds which lead to shorter curing lines and hence reduced cost as well as the ability to cure these coatings on heat-sensitive materials like wood or wood-based materials [11]. The finished products have unique surface characteristics such as good abrasion and solvent resistance and high gloss. In the present study, the acrylated epoxidized linseed oil (AELO) was cured with three different

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Scheme 1. Synthesis of acrylate depoxidized linseed oil (AELO) using acrylic acid monomer from epoxidized linseed oil (ELO).

photoinitiators benzophenone (photoinitiator A); 2,4-diethyl-9H-thioxanthen-9-one (photoinitiator B); 2-hydroxy-2-methyl phenyl propane-1-one (photoinitiator C) at two different concentrations (1 and 2) of each photoinitiator under monochromatic UV light and the cure kinetics of the acrylate were studied from the degree of cure with respect to exposure time using real time FT IR spectroscopy with an ATR unit [12]. The chemical structures of the photoinitiators are given in Fig. 1.

The photoinitiators A and B are of type II while photoinitiator C is an example of a type I photoinitiator systems. Usually, type II photoinitiators are used in addition with co-initiators as a hydrogen donor source which supports the initiation of polymerization. In the current work no coinitiators were used in order to be able to analyze the basic cure kinetics behavior of the photoinitiators without the supporting effect of co-initiators. Whether co-initiators are used or not, type II photoinitiators are normally slower than type I photoinitiators, because the latter generate radical pairs through a highly efficient α -cleavage process [13].

The spruce based wood samples were coated with the newly developed coating system and the coatings were cured under UV light. Finally, the technological properties were tested on the cured surface to determine the degree of cure and to determine the adhesion of the coating on the spruce wood sample.

2. Materials and methods

2.1. Materials

Epoxidized linseed oil (Dehysol B316) with the iodine number of 0–7 g $I_2/100$ g and an oxygen content of 8.5–10% was provided by Cognis, Düsseldorf, Germany. Acrylic acid, triphenylphosphine, hydroquinone, sodium bicarbonate, ethanol, and magnesium sulphate were purchased from Sigma–Aldrich. The photoinitiators benzophenone and 2,4-diethyl-9H-thioxanthen-9-one were purchased from Sigma–Aldrich. The photoinitiator 2-hydroxy-2-methyl phenyl propane-1-one (trade name Darocur 1173) was received as a gift sample from BASF.

2.2. AELO synthesis

AELO was synthesized in the laboratory using the following procedure. Initially, a mixture of epoxidized linseed oil (25 g) and acrylic acid (5 g) containing 0.5 wt% hydroquinone was stirred at a temperature of 75 °C for 20 min. Triphenylphosphine (2 g) was added to the mixture and the mixture was stirred for another 1 h at 60 °C. An additional amount of acrylic acid (4 g) was added to the reaction mixture which was then kept at 60 °C for another 1 h. The consumption of acrylic acid during acrylation was proved by determining the acid number. Unreacted acrylic acid was removed by solvent extraction and the remaining viscous solution was washed with 5% aqueous sodium bicarbonate solution and then with water. Finally, the polymer was dried over magnesium sulphate and the solvent was removed under vacuum distillation.

2.3. Instrumental characterization methods

The structural changes that had occurred after the acrylation process were determined with a FT-IR spectrometer (Bruker Equinox 55) and an attenuated total reflectance (ATR) unit. The IR spectra were recorded as an average of 32 scans in the spectral range between 4000 and 600 cm^{-1} with a resolution of 4 cm^{-1} . The curing characteristics of the three photoinitiators at the monochromatic wavelength of 365 nm were determined using real time FT-IR spectroscopy with an ATR unit. To guarantee comparable UV light intensity absorption by all three photoinitiators in the curing mixtures, the extinction coefficients ϵ_n at 365 nm for known concentrations were measured using a Shimadzu UV 1800 spectrophotometer. In order to determine the extinction coefficients ϵ_n , the photoinitiators were dissolved in ethanol at a concentration of 1 millimole/100 mL and the UV transmission spectra were recorded. For the photo-curing experiments, the photoinitiator concentrations in the AELO resin mixtures were adjusted such that a uniform absorbance value of 0.1 absorbance units was obtained for all three photoinitiators for a film thickness of 20 μm by using the Lambert–Beer relationship:

$$A = \log_{10} \left(\frac{I_0}{I} \right) = \epsilon \times c \times L \quad (1)$$

where A is the absorbance, I_0 is the intensity of the incident light at a given wavelength (i.e., in this case, 365 nm), I is the intensity of the absorbed light, L is the path length through or the thickness of the sample, c is the concentration of the absorbing sample or medium, and ϵ is the extinction coefficient for the given wavelength.

In the AELO mixtures, two concentrations (“high and low levels”) were used for each photoinitiator system (A, B, and C). The photo-initiator concentrations of the high level samples (A2, B2, and C2) were chosen 1.5 times higher than A1, B1 and C1 samples.

The real time FTIR spectra were also recorded using Bruker equinox 55 spectrometer and a heated single reflection diamond Golden Gate ATR unit. The spectra were recorded at a scanning

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