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The Influence of Plasticizer Type on the Film Properties of a Fully-Formulated Aqueous Ethylcellulose Dispersion

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Objectives

Ethylcellulose, a water-insoluble polymeric film-forming material, has been formulated into aqueous latexes (Surelease[®]) for modified released film coating in the pharmaceutical industry.

The purpose of this study was to determine the effect of choice of plasticizer on the dispersion behavior and film properties of a fully formulated aqueous ethylcellulose dispersion.

Methodology

Surelease, an aqueous ethylcellulose latex dispersion, was formulated with oleic acid (OA), or in combination with medium chain triglycerides (OA-MCT), or dibutyl sebacate (OA-DBS), as plasticizers. Dispersions were produced by mixing polymer and plasticizer then melting and extruding the mixture followed by direct emulsification in ammoniated water in accordance with U.S. patent 4,123,403.1 Dispersions were tested for particle size, zeta (ζ) potential, minimum film-forming temperature (MFFT), and rheological properties. Films were cast from the resulting dispersions using a draw-knife casting technique onto Teflon coated glass plates. Cast films were tested for their mechanical properties including tensile strength (σ) and modulus of elasticity (E). In addition, films were also tested for their permeability to moisture and glass transition temperature to assess the effectiveness of plasticizer type and concentration.

Particle size and zeta potential of the dispersions were determined by a Zetasizer Nano-ZS (Malvern Instruments). Dispersion rheology was examined using a stress controlled rheometer AR G2 (TA Instruments).

MFFT of polymer and plasticizer dispersions was assessed using a temperature gradient plate, MFT Thermostair (Coesfeld Material Test, Germany). Testing was conducted in accordance with both ASTM D 2354-91 and ISO 2115 standard methods for determination of minimum film forming temperature or white point temperature of polymer dispersions and emulsions.^{2,3}

The glass transition temperature (T_g) of each formulation was determined using a TA Instruments TMA 2940 Thermomechanical Analyzer (TMA) equipped with an expansion probe to measure thermal coefficient of expansion and glass transition. Samples were tested ($n=3$) under a nitrogen atmosphere (100ml/min) at a heating rate of 10°C/min. The temperature range evaluated was 25 to 180°C.

Free films of all formulations were prepared from aqueous dispersions of 25% solids content using a draw-knife (Gardner Casting Knife, Silver Spring, MD) casting technique onto Teflon[®] coated glass plates. Casting knife settings were kept constant to control the thickness of the wet film in order to obtain the desired film thickness after drying. Cast films were cured at 60°C in an oven for 12 hours and allowed to equilibrate, for a minimum of 24 hours, in a controlled environment laboratory (23°C/55%RH) prior to testing.

The mechanical properties of cast films were determined using a tensile testing instrument (Instron Mini 44 equipped with a Series IX data acquisition and analysis software). Test strips of film were cut into rectangular strips of 10mm x 100mm. The extension rate was 1mm/min. The average ($n=10$) tensile strength and modulus of elasticity were determined for each formulation from the stress-strain curve. The measure of the elastic modulus (E) was taken as the slope of the initial linear portion of the stress (σ) versus strain (ϵ) curve where deformation in this region obeys Hooke's law.

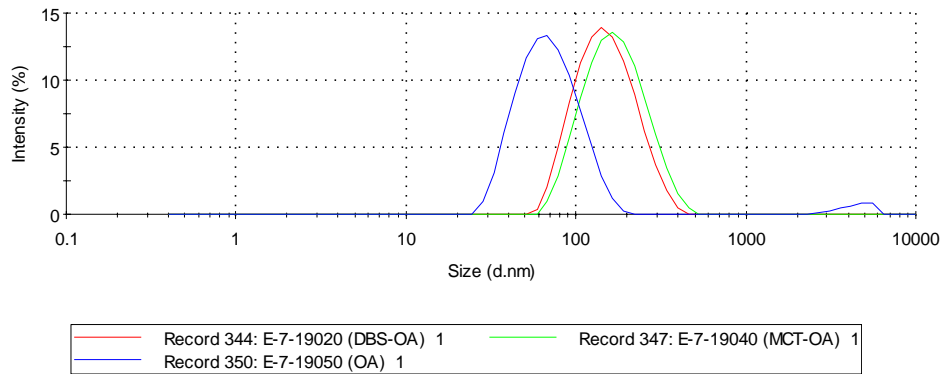
The moisture permeability of the cast films were tested using a WPA-100 water permeability analyzer (VTI Corporation). Experimental test conditions exposed the films to 25°C and 80%RH.

Results

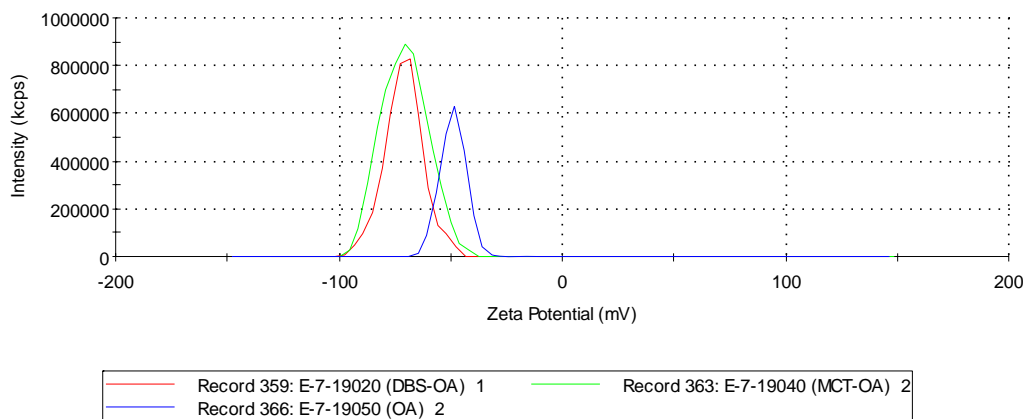


Particle Size & Zeta Potential

Size Distribution by Intensity



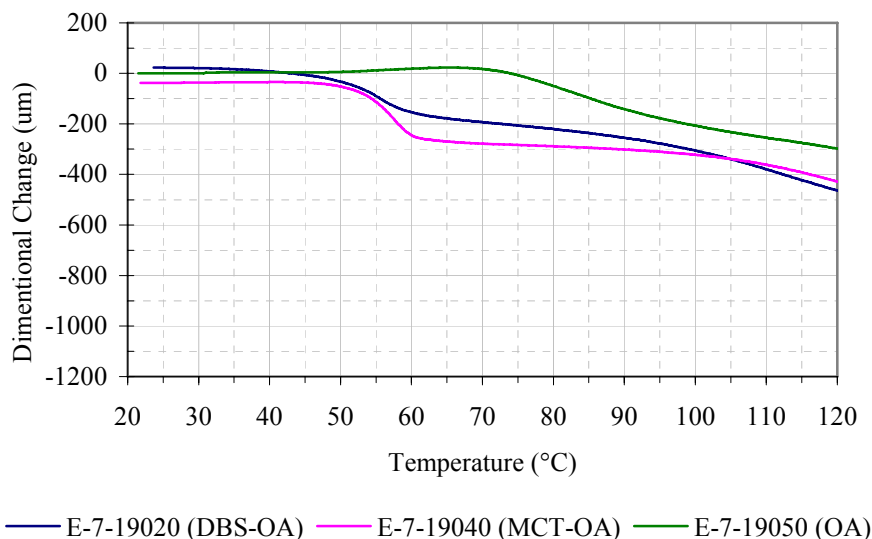
Zeta Potential Distribution



Particle size and ζ -potential are important factors that can affect the stability of aqueous latex dispersions.

Generally, a particle size of less than 500 nanometers and a ζ -potential of greater than ± 30 mV is desirable. Average particle size for the MCT-OA and DBS-OA dispersions was 158 nm and 139 nm respectively. ζ -potential of these systems were both -70 mV. However, the dispersion plasticized with OA possessed a particle size below 100 nm with a ζ -potential of -50 mV.

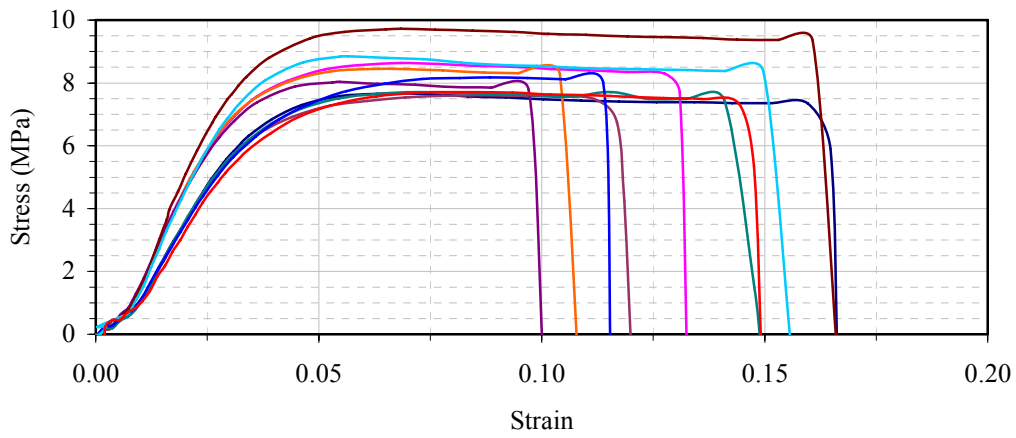
Thermomechanical Analysis & T_g (°C)



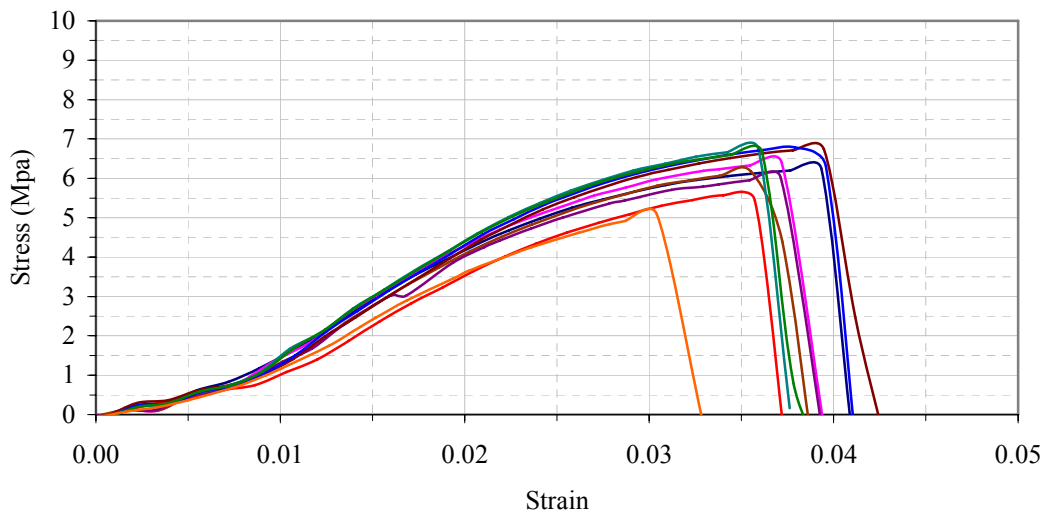
Thermomechanical analysis showed similar onset of Tg for the dispersions plasticized with DBS-OA and MCT-OA while the OA system had a slightly higher transition temperature. Melt behavior of all three systems beyond their Tg was similar.

Tensile Strength & Modulus of Elasticity

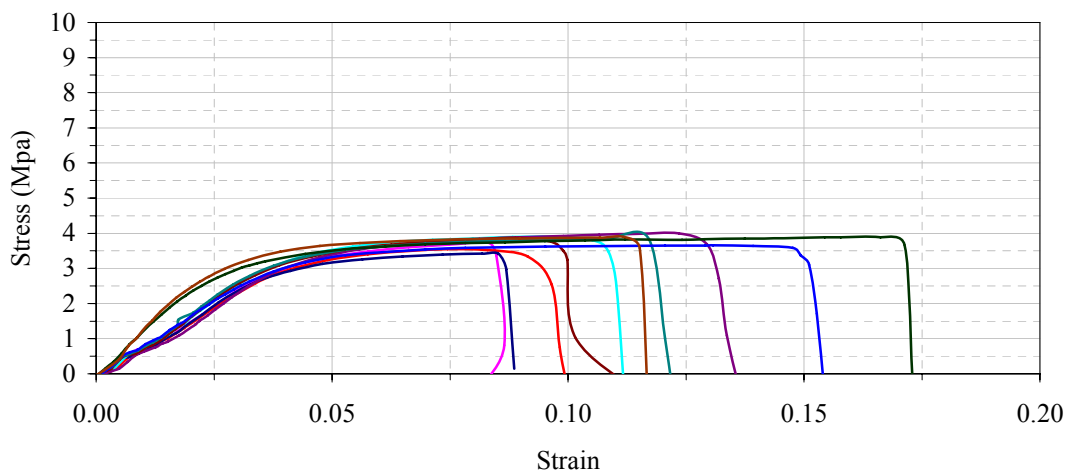
E-7-19040 (MCT-OA)



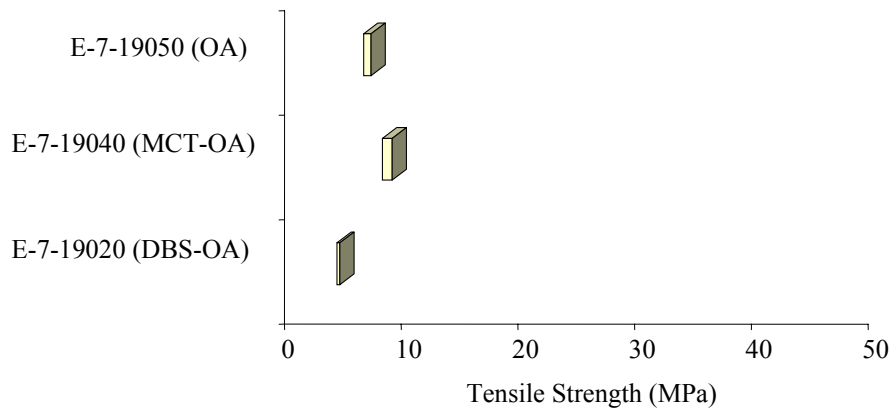
E-7-19050 (OA)



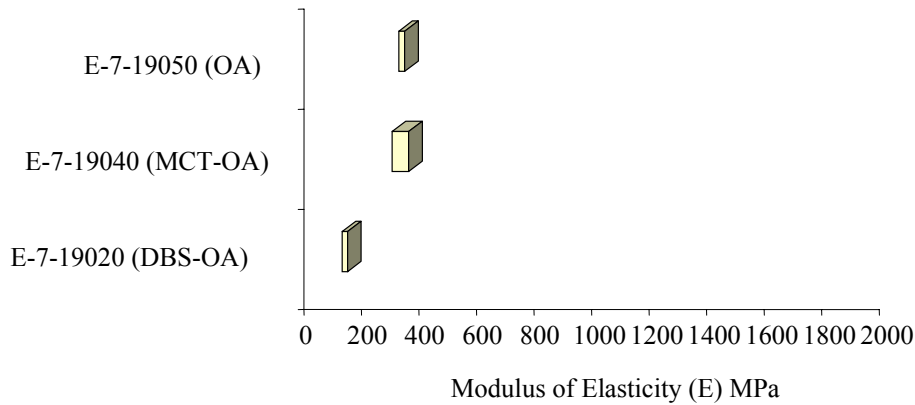
E-7-19020 (DBS-OA)



**Film Properties: Tensile Strength (s) MPa
(90% Confidence)**



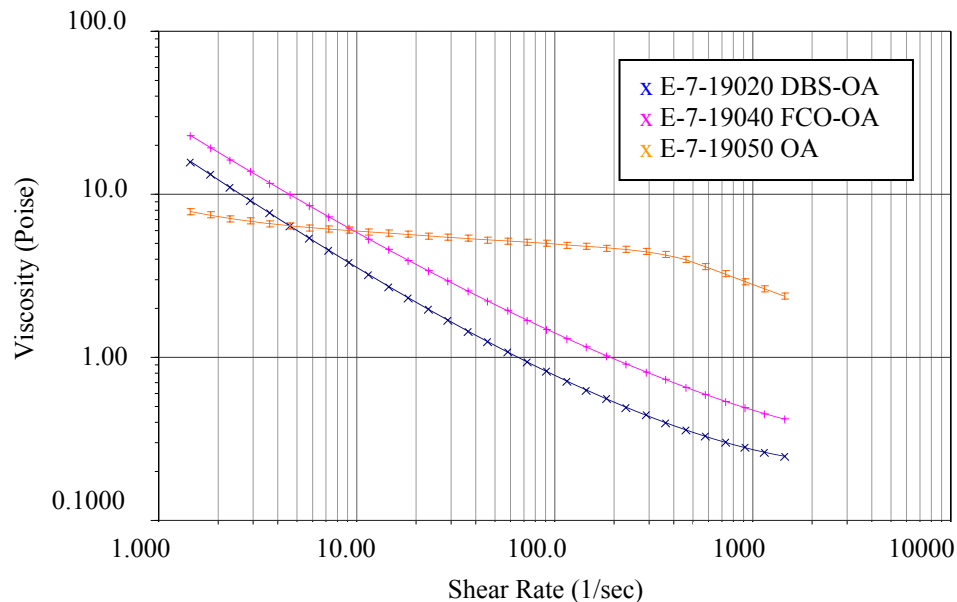
**Film Properties: Modulus of Elasticity (E) MPa
(90% Confidence)**



Stress strain plots (above) indicate that while MCT-OA films possessed similar tensile strength to OA films, the MCT-OA films were more ductile in behaviour. However, DBS-OA cast films showed lower overall tensile strength and modulus of elasticity.

Rheology

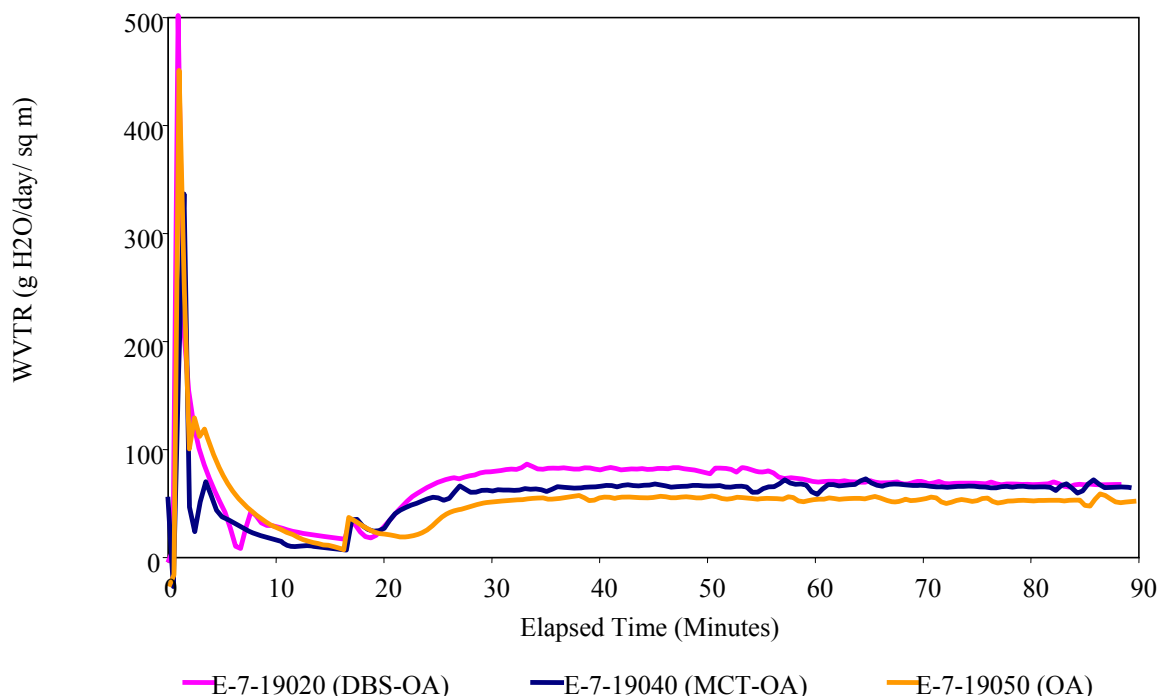
Steady State Flow Test



Rheological characterization showed that while the dispersions plasticized with DBS-OA or MCT-OA exhibited shear dependence over a broad range of shear rates the dispersion plasticized with OA showed less of a shear dependence except when approaching shear rates of greater than 1000 sec⁻¹

Permeability

Water Permeation Analysis of Cast Films - E-7-190X0 25°C / 80% RH



At equilibrium, the moisture permeation rate of DBS-OA and MCT-OA films were similar at approximately 97gmH₂O/day/m², whilst the OA system had an average permeation rate of 82gmH₂O/day/m².

Conclusion

The thermal and physical properties of a fully formulated aqueous ethylcellulose dispersion, containing different plasticizers, were evaluated. Dispersions, with OA, OA-MCT or OA-DBS as plasticizer, resulted in particle size less than 200 nanometers. Physical properties of the dispersions and thermal behavior of films, cast from the dispersions, differed only slightly despite the different choices of plasticizer.

References

1. US Patent 4,123,403 December 1982 by Leng et al of Dow Chemical Company *Aqueous Dispersion Of Plasticized Polymer Particles*.
2. ASTM D 2354-91, Standard Method for Minimum Film Formation temperature (MFFT) of Emulsion Vehicles. (Re-approved 1997)
3. International Standard (ISO) 2115, Plastics - Polymer dispersions - Determination of white point temperature and minimum film-forming temperature.

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