Spectroscopic Investigations of Phase-Separated Thermotropic Layers Based on UV Cured Acrylate Resins

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Summary: In this paper spectroscopic techniques were adapted and applied to characterize the optical and morphological properties of thermotropic resins. Thermotropic films were prepared by variation of resin base and thermotropic additives. By UV/Vis/NIR spectroscopy the solar optical properties, the switching temperature, the switching process and the residual transmittance in the opaque state were determined. To control the sample temperature from ambient to 100 °C a conventional UV/Vis/NIR spectrophotometer equipped with an Ulbricht-sphere was adapted by a heating stage. Additive content, distribution and scattering domain size were characterized by Differential Scanning Calorimetry, Attenuated Total Reflectance spectroscopy and microscopic techniques. For the investigated film types hemispheric solar transmittance values ranging from 80 to 87% and 75 to 85% were obtained in the clear and the scattering state, respectively. The diffuse solar transmittance values increased significantly from 14 to 40% below the switching temperature to 36 to 70% at elevated temperatures. The thermotropic resins exhibit a steep and rapid switching process with switching temperatures between 45 and 70 °C. The materials with the best switching properties are characterized by distinct differences in refractive index between the components, a uniform additive distribution and scattering domains with diameters below 1000 nm.

Keywords: infrared spectroscopy; overheating protection; refractive index; thermotropic resins; UV-Vis spectroscopy

Introduction

Polymeric materials are playing a key-role in the future development of solar energy systems, as they offer significant potential for cost savings in solar thermal collectors and may thus benefit a broader utilization of solar energy, specifically for low-temperature heating purposes. Especially for domestic hot water generation and space heating applications, where the required maximum operating temperature of the absorber is about 80 °C, cost-efficient plastics will be applied. However, conventional solar thermal collectors reach stagnation temperatures up to 200 °C, which exceed the maximum operating temperatures of cost-efficient plastics (~80–140 °C). Absorbers made of such plastics tend towards irreversible deformation and/or degradation when exposed to elevated temperatures of 200 °C for prolonged periods. Therefore an appropriate overheating protection is required. The energy flux in an all-polymeric flat-plate collector can be controlled by thermotropic layers.[1,2] Thermotropic materials change their light transmission behavior from highly transparent to light diffusing upon reaching a certain threshold temperature.[3,4] Thus such actively switchable layers permit the light and energy flux to be adapted dynamically to climatic

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demands. By theoretical modeling of an all-polymeric flat plate collector it was found that for adequate overheating protection (i.e. stagnation temperatures ranging from 80 to 140 °C) switching temperatures between 55 and 60 °C as well as a residual hemispheric solar transmittance of 25 to 60% in the opaque state are effectual.[1]

In thermotropic materials that undergo a transition from transparent to light diffusing, the light is scattered from particles, which exhibit an index of refraction, which is different to that of the matrix.[3,4] In the past various thermotropic systems for active daylight control in transparent facades have been developed and investigated.[3–10] Whereas in thermotropic polymer blends and hydrogels the switching is involved in a phase separation, in thermotropic resins, the scattering domains are embedded statically in a matrix material.[3] To the best of our knowledge, comprehensive and systematic development and research on thermotropic materials for solar thermal systems has not yet been performed. Hence, the overall objective of our research is to perform a comprehensive development and characterization of thermotropic materials for overheating protection of solar collectors and to establish structure-property relationships. Various thermotropic resins were manufactured by systematic variation of base resin formulation as well as of additive type. The materials are characterized as to their optical properties and switching characteristics on a UV/Vis/NIR spectrophotometer equipped with an Ulbricht-sphere to differentiate between diffuse and direct transmitted light. Additionally the indices of refraction are considered. Scattering domain size, additive content and distribution of the thermotropic resins are characterized applying Differential Scanning Calorimetry, light microscopy and Attenuated Total Reflectance spectroscopy.

Material Preparation

Thermotropic layers were prepared by dissolving the thermotropic additives in a photo-crosslinkable matrix solution, which consists of oligomers, a reactive diluent as well as a photoinitiator. For the present study 2 different oligomer systems (oligomers A and B) serving as the main component of the matrix material were selected. In order to meet the requirements as to overheating protection of solar thermal systems, 3 thermotropic additives exhibiting switching temperatures of 47, 54 and 60 °C were selected (additive types 1, 2 and 3).[1] The additive concentration chosen for the preparation of the films was 5 wt%. The dissolution of the thermotropic additive in the resins was filled into an intervening space located between two glass panes, which were sealed around the edge. Afterwards the mixture was cured by UV-radiation (Ultra Vitalux, Osram GmbH, München, GER) for 5 minutes. The film thickness was 800 μm. As to the nomenclature A-1 indicates a film based on resin A containing additive type 1, for example.

Characterization of Optical Properties

The refractive indices of the additives and the resins were determined on a m-line apparatus (Metricon model 2010 (Metricon Corp., Pennington, USA)) based on the prism coupling technique. All measurements were performed at temperatures from 25 to 100 °C using a He-Ne laser with a wavelength of 633 nm.

The thermotropic materials were analyzed as to solar transmittance values in clear and opaque state, switching temperature and switching process applying UV/Vis/NIR spectroscopy. Hemispheric and diffuse transmittance spectra were recorded at normal incidence within a wavelength range between 250 and 2500 nm on a conventional UV/Vis/NIR spectrophotometer (Lambda 950, Perkin Elmer Instruments GmbH, Überlingen, GER) equipped with an Ulbricht-sphere (diameter 150 mm). For the given measurement apparatus the radiation passing through the specimen outside a cone of approximately 5° relative to the incident beam direction is defined as diffuse (scattered) component.

The measured spectral data were weighted in steps of 5 nm by the AM 1.5 global solar irradiance source function, given in Bird and Hulstrom[11] and representing the spectral distribution of solar radiation energy, when the sun is at approximately 42° above the horizon at sea level where the solar radiation has traveled 1.5 atmospheres. By this way integral solar transmittance values were determined.

To investigate the switching behavior (i.e. switching characteristic and temperature) the spectrophotometer was adapted by a heating stage allowing for controlling the sample temperature from ambient to 100 °C. For the determination of films thermal transitions the samples were mounted in a heating-chamber within the standard sample chamber of the spectrophotometer using a purpose-built clamp. This clamp has been additionally adapted by sensors to acquire the temperatures of the chamber and the sample. Due to this setup the sample is not positioned directly at the entrance of the integrating sphere. Especially for highly diffuse scattering samples this causes significant chamber recess error, which in turn results in considerable deviations between the recorded values and the effective radiation transmitted by the sample. To avoid these radiative losses on the one hand the inner boundaries of the light transmitting recess in the heating chamber were covered with a highly reflective mirror film which exhibits direct-direct and direct-diffuse solar reflectance values of 80 and 5%, respectively. On the other hand, the dimension of the incident light beam striking the sample has been reduced by a pinhole that was mounted at the front side of the heating chamber. With this configuration even for highly diffuse scattering samples more accurate transmittance values were measured. However, slight differences between the detected values and the effective radiation transport were obtained, which can be attributed to absorption of the mirror film (merely 85% solar reflectance). Thus a calibration of the recorded data is required. For this purpose hemispheric and diffuse transmittance spectra of numerous weak and highly scattering samples were recorded both by positioning the samples direct within the portholes of the Ulbricht-sphere and by mounting the materials in the adapted heating stage. In Figure 1 hemispheric and diffuse solar transmittance values, recorded at the different sample positions are compared. Excellent linear correlations with regression coefficients of 0.99 and 0.96 for hemispheric and diffuse solar transmittance values, respectively,

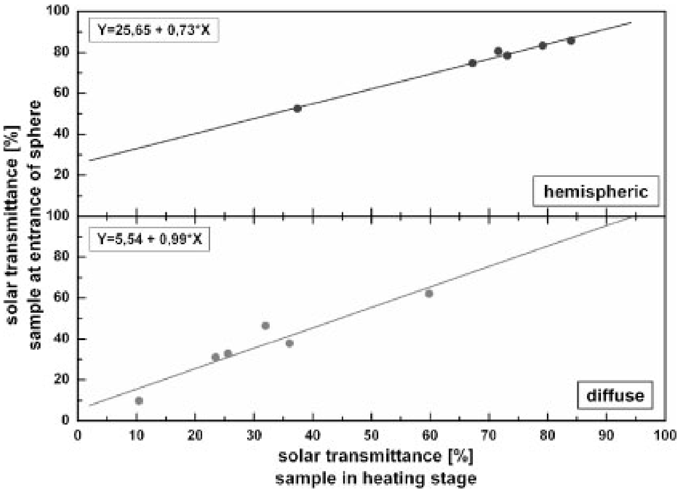

Figure 1.
Calibration for hemispheric and diffuse transmittance.

were obtained. All data presented in the following have been corrected by the corresponding equations of the linear regressions given in Figure 1.

Transmittance measurements were performed in steps of 5 K. Prior to the measurements the sample was maintained at the selected temperature for 10 minutes.

Characterization of Morphology

The additive contents of the produced thermotropic layers were determined by Differential Scanning Calorimetry operating in Temperature Modulation mode (TM-DSC). TM-DSC was performed under static air from 0 °C to 130 °C at a heating rate of 1 K/min, a modulation amplitude of ±0.5 °C and a modulation period of 75 s on a Mettler Toledo DSC822e (Schwarzenbach, CH). Weighting the transition enthalpy of the thermotropic layer by the heat of fusion of the pure additive allowed for the evaluation of the films additive content.

To obtain information as to film composition and additive distribution on the surface of the thermotropic materials Fourier Transform Infrared (FT-IR)-spectroscopy was performed in Attenuated Total Reflection mode (ATR) within a wavenumber range from 700 to 4000 cm^{-1} at a resolution of 1 cm^{-1} using a diamond crystal (Spectrum One, Perkin Elmer Instruments GmbH, Überlingen, GER). The recorded spectra were corrected for the wavelength-dependent penetration depth of the evanescent wave as well as for the baseline by the *Spectrum v. 5.0* software (Perkin Elmer Instruments GmbH, Überlingen, GER).

Additive particle sizes were characterized applying optical microscopy. Transmitted light micrographs were recorded on an Olympus BX51 (Olympus GmbH, Hamburg, GER) microscope at a magnification of 50×.

Results and Discussion

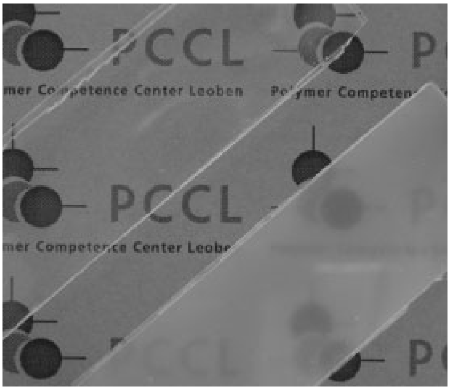

Figure 2.
Thermotropic layer A-1 in clear (left) and scattering state (right).

In Figure 2 a representative thermotropic resin (layer A-1) in transparent and light diffusing state is depicted. The thermotropic material is perfectly clear below the switching temperature (left side). At elevated temperatures the light scattering of the sample increases, which causes the material to turn opaque.

In Figure 3 to 6 hemispheric and diffuse transmittance spectra of the different thermotropic layers based on the resins A and B in the clear and scattering state are depicted, respectively. In the transparent state (solid line) all layers show a hemispheric transmittance of about 80% in the visible and the near-infrared region (Figure 5 and 7). No significant differences between the various materials are apparent. Interactions between the films and the solar radiation which reduce transmission occur below 380 and above 1200 nm. Below 380 nm the transmittance decreases due to absorption of incorporated ultraviolet absorbers. Absorption bands at approximately 1200, 1400 and 1700 nm and above 2225 nm can be attributed to vibrations of carbon-hydrogen single bonds (C–H). The peak at 2150 nm appears due to stretching of carbon-oxygen double bonds in the polymers (C=O). The absorption peak at about 1900 nm refers to water molecules absorbed by the material. In the scattering state (dotted line) material B-1 displays slightly higher transmittance values compared to the clear state. For the other layers

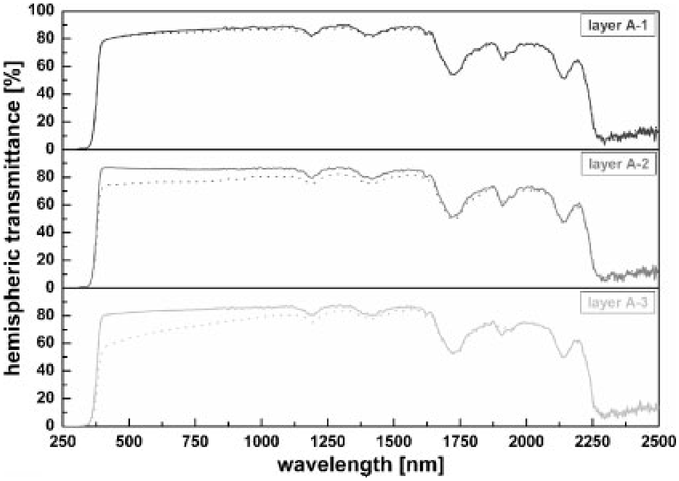

Figure 3.
Hemispheric transmittance spectra of thermotropic layers based on resin A in clear and scattering state; top: layer A-1; mid: layer A-2 and bottom: layer A-3 (solid line: clear state; dotted line: scattering state).

a decrease of the hemispheric transmission is discernible at elevated temperatures. In general these layers show a stronger decrease of transmission in the region between 300 and 1500 nm than at longer wavelengths. The differences in transmission between the clear and the scattering state increase with decreasing wavelength. The reduction of transmittance is more pronounced for materials A-2, A-3 and B-3. This indicates that these layers exhibit better light shielding properties than film types A-1, B-1 and B-2.

The diffuse transmittance spectra of the investigated thermotropic materials shown in Figure 4 and 6 give information as to their scattering properties. The spectra reveal significant differences in the film's scattering behavior. All film types exhibit an increase of diffuse transmittance with

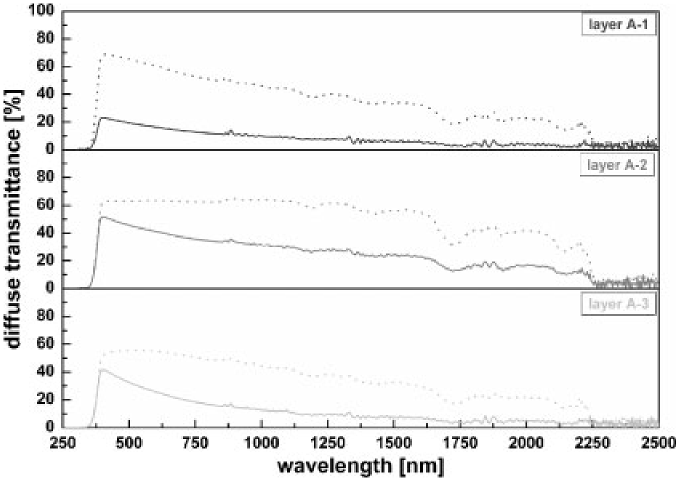

Figure 4.
Diffuse transmittance spectra of thermotropic layers based on resin A in clear and scattering state; top: layer A-1; mid: layer A-2 and bottom: layer A-3 (solid line: clear state; dotted line: scattering state).

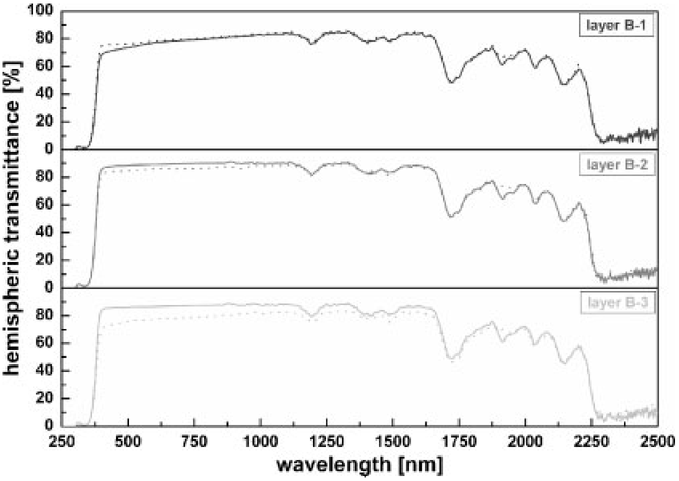

Figure 5.
Hemispheric transmittance spectra of thermotropic layers based on resin B in clear and scattering state; top: layer B-1; mid: layer B-2 and bottom: layer B-3 (solid line: clear state; dotted line: scattering state).

decreasing wavelength. Whereas additive type 1 yields lower diffuse scattering for layers based on resin A, additives types 2 and 3 reduce the diffuse scattering power for layers produced on the basis of resin B in the clear state (solid line). For the films A-1, B-2 and B-3 the diffuse transmittance values were found to remain below 30% over the whole wavelength range only slightly increasing with decreasing wavelength. For the layers A-2, A-3 and B-1 a more pronounced increase of the diffuse transmittance values in the short wavelength range was observed associated with also higher diffuse transmittance values in the near infrared wavelength range. In the scattering state (dotted line) a significant increase of diffuse transmittance is discernible in the wavelength range between 380 and 2250 nm. The most pronounced rise

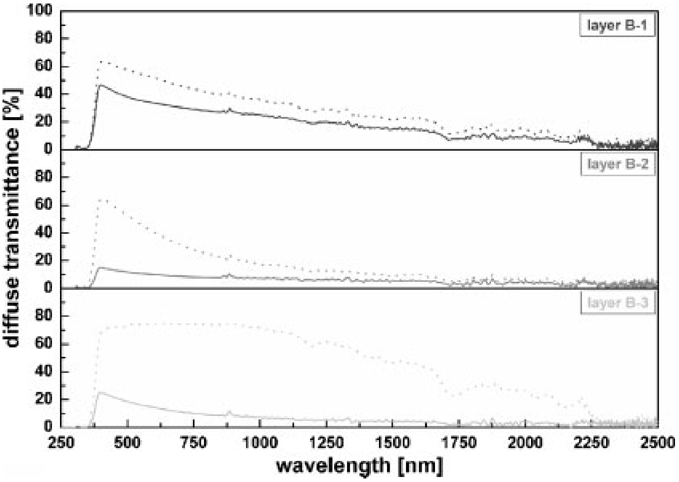

Figure 6.
Diffuse transmittance spectra of thermotropic layers based on resin B in clear and scattering state; top: layer B-1; mid: layer B-2 and bottom: layer B-3 (solid line: clear state; dotted line: scattering state).

of diffuse scattering power was found for the layers A-1 and B-3. Layer B-1 exhibits the less distinct changes. The differences in diffuse scattering below and above the switching temperature increase with decreasing wavelength for layers A-1, B-1 and B-2. For the layers showing the most significant reduction of hemispheric transmittance (layers A-2, A-3, B-3) this tendency is not observable.

The hemispheric and diffuse transmittance spectra of the samples clearly indicate that the thermotropic layers undergo a transition from a clear to a scattering state. Accurate relationships between material formulation and the scattering behavior may be emphasized by integral solar optical properties determined over the solar spectral intensity AM 1.5. Thus in Table 1 integral solar optical properties (hemispheric and diffuse transmittance) of the investigated thermotropic layers in the clear and the opaque state are summarized. Below the switching temperature all samples exhibit an excellent hemispheric transmittance above 80%. For the diffuse transmittance considerable differences between the various material formulations are observable. The values range from 14 to 40%. Whereas for layers based on resin A additive type 1 yields lower diffuse scattering, materials with resin B matrix show better optical properties with the additive types 2 and 3. In the scattering state at elevated temperatures the diffuse transmittance increases significantly to values exceeding 36%. The most distinct changes are observable for the layers B-3 and A-1, where the forward scattered light increases by 53 and 37%. The diffuse transmittance of the other layers increases by 13 to 26%. Already a change of the diffuse transmittance of 13% is associated by a loss of view-through behavior. However, the overheating protection properties are determined by the reduction of hemispheric transmittance. The hemispheric transmission of the films is decreased above the switching temperature to a smaller extent. The most significant changes were found for thermotropic layers being formulated with additive type 3. By dispersing this additive type in the resins A and B the hemispheric transmittance is reduced by 6 and 9%, respectively. A reasonable decrease of the hemispheric transmission was also obtained for material A-2 (6%). For material B-1 an increase of transmittance from 80 to 81% is discernible. This layer exhibits a significant increase of transmittance to values of 85% at temperatures between 30 and 45 °C.

To describe the switching characteristics of the materials in Figure 7 and 8 the diffuse solar transmittance is plotted as a function of temperature for thermotropic materials based on resin A and B, respectively. For thermotropic layers being formulated with additives 1 and 2 the switching gradient is reasonably steep, so that the diffuse solar transmittance changes within a small temperature range of 10 to 15 K. The temperature at which this transition occurs (switching temperature) ranges between 40 and 50 °C for these film types. Whereas for the thermotropic layer produced from

Table 1.
Hemispheric and diffuse solar transmittance values of the thermotropic layers in clear and scattering state.

Resin	Additive type	Solar transmittance			
		Clear state		Scattering state	
		Hemispheric [%]	Diffuse [%]	Hemispheric [%]	Diffuse [%]
A	1	85	19	84	56
	2	85	40	79	64
	3	84	25	75	51
B	1	80	34	81	47
	2	87	14	85	36
	3	86	17	80	70

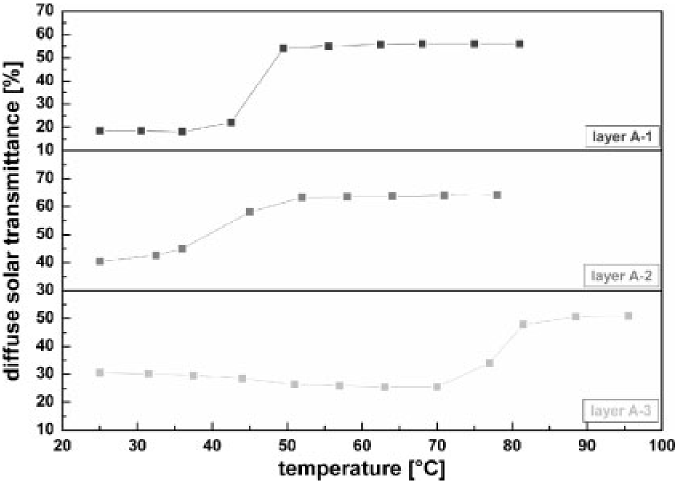

Figure 7.
Diffuse solar transmittance of thermotropic layers based on resin A as a function of temperature; top: layer A-1; mid: layer A-2 and bottom: layer A-3.

additive type 3 and resin B the switching again proceeds at a temperature of about 45 °C, the transition from the clear to the scattering state is more gradual. In contrast the thermotropic layer produced from resin A and additive type 3 shows a very steep switching process. However, the switching temperature of this film is shifted to above 70 °C.

Both, the scattering spectra and the solar optical transmittance data presented in Table 1 reveal significant differences in the scattering behavior between the various thermotropic layers. In general the optical properties and the switching behavior of the thermotropic materials are determined both, by the differences in refractive index between the additives and the matrix

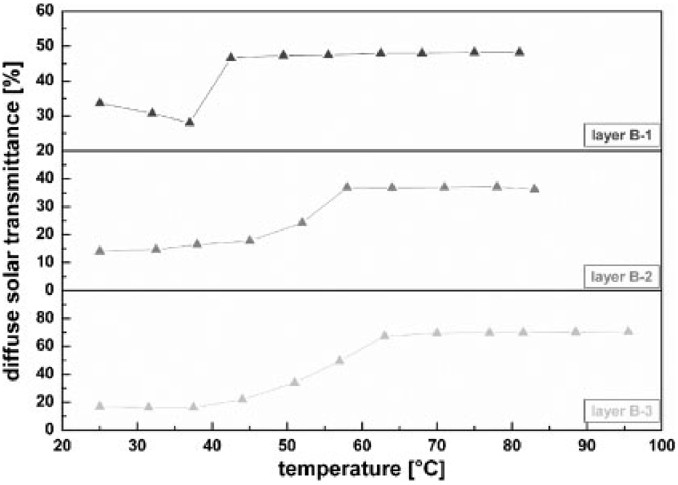

Figure 8.
Diffuse solar transmittance of thermotropic layers based on resin B as a function of temperature; top: layer B-1; mid: layer B-2 and bottom: layer B-3.

material below and above the switching temperature, and by the scattering domain size and number of scattering particles, which is correlated to additive content.[12] The consideration of these parameters is of significant importance for the establishment of a fundamental understanding of the layers switching characteristics and a systematic material development and optimization.

In Figure 9 the refractive indices of the additives and the matrix materials are compared at different temperatures. For the resins A and B refractive indices of 1.52 and 1.50 were determined at low temperatures, respectively. The insignificant decrease of 1.1 (resin A) and 1.3% (resin B) in the temperature range between 25 and 95 °C indicates that the refractive index of the resins is not affected by changes in temperature. The refractive index of additive type 1 switches steeply from 1.52 to 1.45 within 40 and 50 °C. For additive type 2 the refractive index decreases from 1.50 to 1.42 in the temperature range from 25 to 50 °C. For additive type 3 the decrease of the refractive index is more gradual. It changes from 1.52 to 1.43 in a temperature range between 30 and 90 °C.

In general below the switching temperature a reasonable match of the refractive indices of the resins and the thermotropic additives with differences between 0 and 0.02 was detected. Whereas hemispheric transmittance values were found to be insignificantly affected by the differences in the refractive index of the components, the diffuse transmittance varies between the different film types. An increasing diffuse transmittance with increasing difference in refractive index is observable. Figure 9 shows that the differences in the refractive indices of the components at elevated temperatures are more pronounced for the additive types 2 and 3 compared to additive type 1. This may be attributed to a more distinct change in the refractive index of the additive types 2 and 3. However, at elevated temperatures no correlations between the differences in refractive index and the scattering properties of the samples are observable. This indicates that the optical properties of the considered thermotropic layers are further dependent on the additive concentration and distribution as well as on scattering domain size.

As to additive content significant deviations from the theoretical concentration were ascertained, especially for the resins formulated with additive types 2 and 3. For additive type 2 dispersed in the resins A and

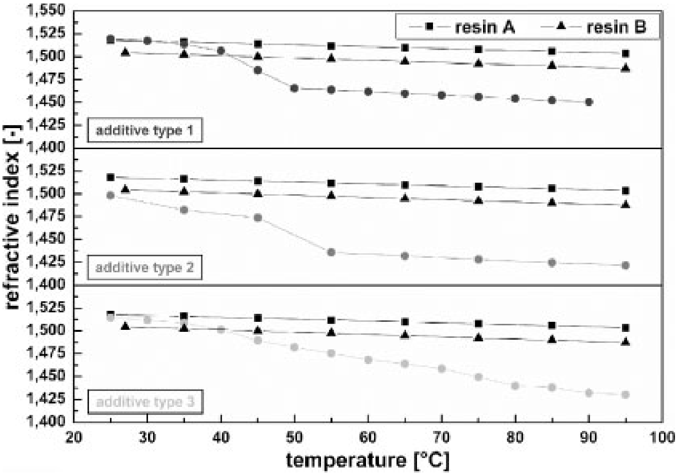

Figure 9.

Refractive indices of resins A and B and the thermotropic additives at a wavelength of 633 nm as a function of temperature; top: resins vs. additive type 1, mid: resins vs. additive type 2 and bottom: resins vs. additive type 3.

B the additive content amounts to 1.33 and 1.60 wt.%, respectively. Scattering domain concentration in films formulated with additive type 3 is even lower with 0.95 and 1.09 wt.% for layers based on resin A and B. The highest additive content was found for thermotropic materials containing additive type 1 with values of 3.87 and 4.80 wt.% in layers A-1 and B-1.

In Figure 10 and 11 ATR spectra in the wavenumber range from 3050 to 2800 cm^{-1} of the various thermotropic layers and the neat resins are depicted. For the thermotropic materials spectra are shown both, for the front side, i.e. the side faced to the UV light source during the curing and for the opposite side, i.e. the back side of the films. Information as to additive concentration on the surface can be deduced from the decrease of transmission at wavenumbers of 2848 and 2915 cm^{-1}, which can be attributed to symmetric and asymmetric stretching vibrations of methylene groups (–CH_2–), respectively. In general the absorption bands are more pronounced for thermotropic layers produced from additive type 1 than for materials formulated with the additive types 2 and 3. This can be ascribed to the higher additive concentration of layers containing additive type 1. Whereas for thermotropic materials based on the additive types 2 and 3 no differences between the two film sides are observable, additive concentration is higher on the front surfaces of layers formulated with additive type 1. It is observable that the absorption peaks are more distinct for thermotropic materials based on resin B. On the one hand this can be attributed to the slightly higher additive content of these materials. On the other hand it may be an indication for a less uniform additive distribution across the film thickness.

The scattering domain sizes of the thermotropic layers were determined applying light microscopy. For materials based on resin A and formulated with the additive types 1, 2 and 3 average particle diameters of 1.7, 1.0 and 0.8 μm were obtained, respectively. Thermotropic layers based on resin B and formulated with additive types 1, 2 and 3 exhibit scattering domains with diameters of 1.4, 2.2 and 1.1 μm, respectively. In

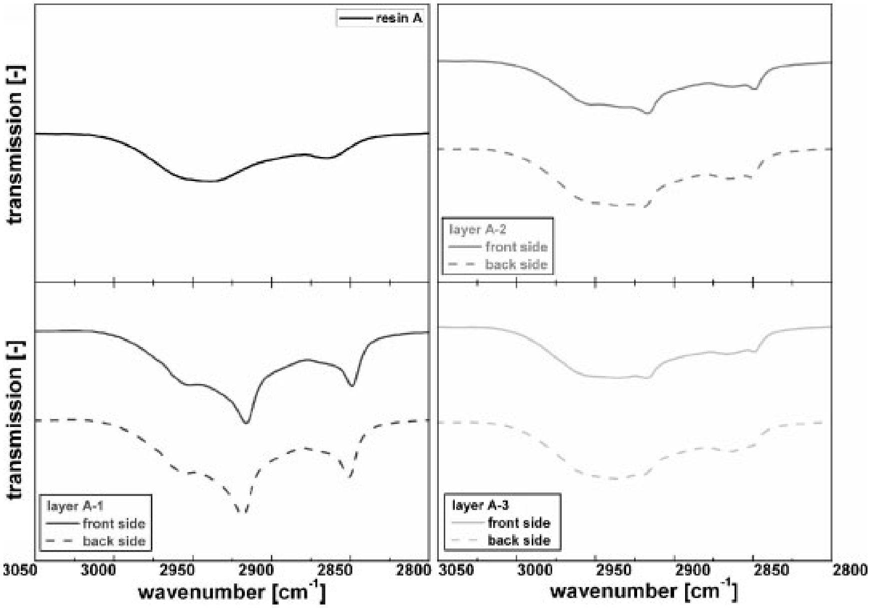

Figure 10.

ATR spectra of resin A and the thermotropic layers based on resin A in the wavenumber range between 3050 and 2800 cm^{-1}; top left: resin A, bottom left: layer A-1, top right: layer A-2 and bottom right: layer A-3.

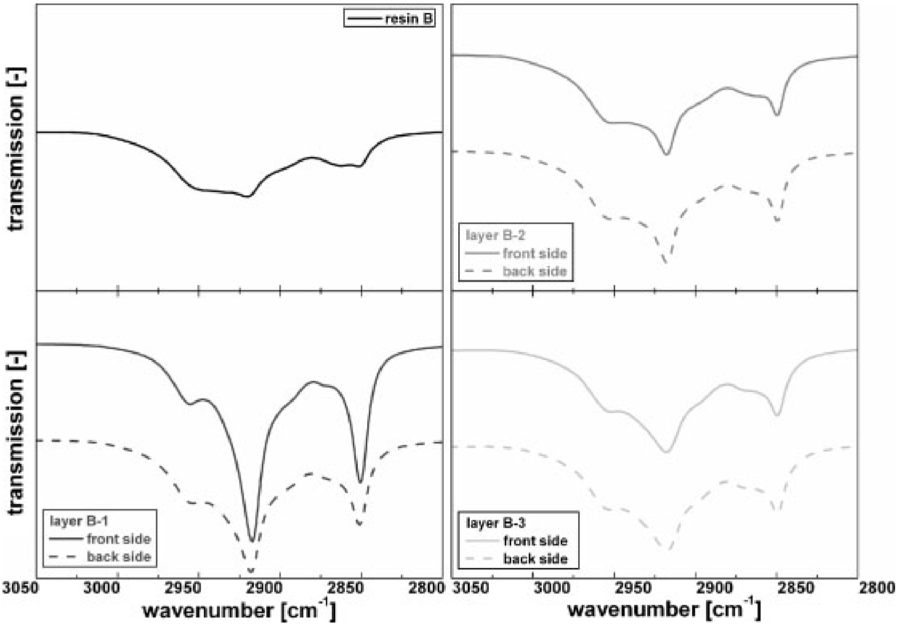

Figure 11.
ATR spectra of resin B and the thermotropic layers based on resin B in the wavenumber range between 3050 and 2800 cm^{-1}; top left: resin B, bottom left: layer B-1, top right: layer B-2 and bottom right: layer B-3.

general a tendency was observed that with decreasing particle sizes the light shielding properties of the thermotropic layers are enhanced. Nevertheless, for maximum reflectance efficiency in the solar wavelength range scattering radii of 100 to 200 nm are required.[5,6,12,13] The scattering domain sizes of the thermotropic materials produced within the present study are significantly larger, which results in less light-shielding capability.

Furthermore the micrographs of the materials depicted a higher aggregation of additives for materials based on resin B. This is convenient with ATR spectra, revealing higher additive concentrations on the surface for these materials. For layer B-3 the additives are less pronounced on the surfaces. Because light microscopy does not allow for a detailed analysis of scattering domain size distribution further investigations are currently carried out using Atomic Force Microscopy (AFM).

As to the influence of morphological parameters on the optical properties of the films it was found that although additive concentration is higher for thermotropic layers based on resin B, both the forward and the back scattering power above the switching temperature is higher for materials produced from resin A. This may be ascribed to the more uniform additive distribution across the film thickness for these materials. The decreased hemispheric solar transmittance of the layers A-1, A-2 and B-3 above the switching temperature can be attributed to the smaller scattering domain size of these films. In general a tendency is observable that decreasing scattering domain size increases the diffuse scattering power. Furthermore a more uniform distribution of the additives across the film thickness yields better light-shielding properties, and thus higher opacity in the scattering state.

Summary and Conclusion

In this paper the optical and morphological properties of thermotropic resin materials are described and discussed. In the clear state the investigated films exhibit direct-hemispheric solar transmittance values of 80 to 87%, with diffuse fractions ranging from 14 to 40%. The materials undergo a transition from the clear to the scattering state at temperatures between 40 and 75 °C. The investigated thermotropic resins exhibit a steep and rapid switching process. Above the switching temperatures the hemispheric solar transmission decreases to values between 75 to 85%, with diffuse fractions ranging from 36 to 70%. The materials show a stronger decrease of transmittance in the visible than in the near infrared region.

The optical properties of the films in the clear and the scattering state were found to be significantly dependent on the differences in the refractive indices of the components, additive content and distribution as well as on scattering domain size. The light-shielding properties of the thermotropic layers and thus the opacity in the opaque state are enhanced by more pronounced differences in refractive index between matrix and additive (>0.04), a uniform additive distribution across the film thickness and scattering particles with dimensions below 1000 nm.

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