

Research Article

Structural and Surface Characterization of Newly Synthesized D- π -D Type Schiff Base Ligand: (1E,2E)-3-[4-(Dimethylamino)phenyl]prop-2-en-1-ylidene)phenylamine

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A new Schiff base with D- π -D type, (1E,2E)-3-[4-(dimethylamino)phenyl]prop-2-en-1-ylidene)phenylamine, has been successfully synthesized using the reaction of (2E)-3-[4-(dimethylamino)phenyl]acrylaldehyde with aniline. The Schiff base ligand has been characterized by FTIR, UV-visible, and ^1H NMR as well as TG/DTA, SEM, BET, and elemental analyses and mass spectra. Surface properties and acid-base constants of Schiff base ligand were determined by IGC measurements.

1. Introduction

The coordination compounds including the Schiff base ligands are of significantly important and play a pivotal role in industry, technology, and life processes. Due to their potential applications in various fields, it has always fascinated and inspired chemists in the world. This can be evidenced by the vast prolificity and scope of research papers on the subject in recent times and also by the diversity in which it has found applications. Some Schiff bases and their derivatives represent an interesting class of compounds possessing a wide spectrum of biological activities, such as analgesic, antiviral, antifungal, and anticancer activities. The Schiff base ligands and their complexes have been a fascinating area of research, due to their biological relevance. These compounds can act as pro- or antioxidants. The published opinions on the structure and antioxidant activity relationships are, however, quite inconsistent. It can be assumed from several scientific works that the quality of antioxidant action depends on the type of ligands forming the bioactive complexes [1, 2].

In the past few decades, very fast development in the field of organic-based conjugated materials was observed [3, 4]. Schiff bases and their metal complexes have many applications in medicinal, industrial, pharmaceutical, biological, and chemical fields. For example, they are industrially

used as dyes and pigments in textile besides their application in pharmacology for the synthesis of antibiotics, antiallergic, and antitumor substances. The Schiff bases can also exhibit thermochromic and photochromic properties [5–7]. Moreover, the Schiff base compounds have been regarded as excellent fluorescent materials because of their ability to achieve high thermal stability as well as high photoluminescent efficiency. The conjugated Schiff base systems that contain electronically coupled photo- and/or redox-active sites across an unsaturated organic bridge are of considerable current interest [8–13].

Many complicated factors can affect the two-photon excitation (TPE) properties of organic materials. So it is important to investigate the structure and the TPE effect relationships of organic materials. However, most of these compounds employ C=C bonds as the conjugation bridge; the compounds with C=N structure such as the Schiff bases are less studied to our knowledge [14, 15].

In the present work, a new Schiff base ligand with D- π -D type which is N,N-dimethyl-4-[(1E,3E)-3-phenylimino]prop-1-en-1-yl]aniline{(1E,2E)-3-[4-(dimethylamino)phenyl]prop-2-en-1-ylidene)phenylamine (SBL) has been successfully synthesized using the reaction of (2E)-3-[4-(dimethylamino)phenyl]acrylaldehyde with aniline.

It can be used as a pigment in some applications because of excellent yellow colour. The structure of the SBL was determined by elemental analysis, FTIR, UV-visible, ^1H NMR, TG/DTA, and LC-MS spectrometry. In addition, IGC was applied to the investigation of adsorption properties of the SBL. The relation of SBL with used polar and nonpolar solvents was tested at temperatures between 30°C and 60°C under melting point of SBL. The goals of this paper were (1) to synthesize and characterize new D- π -D type Schiff base ligand, (2) to figure out the surface free energy of SBL, (2) to calculate adsorption thermodynamic parameters of SBL, (3) to determine the acid-base quantity of SBL.

2. Experimental

2.1. Materials. All reagents and solvents were of reagent grade quality, obtained from commercial suppliers (Merck, USA). Aniline and (2E)-3-[4-(dimethylamino)phenyl]acrylaldehyde were used as supplied commercially from Merck. The progress of the reaction was monitored by TLC (SiO_2). IR spectra were recorded on a Perkin Elmer Spectrum One FTIR (ATR sampling accessory) spectrophotometer; electronic spectra in the UV-Vis region were recorded with an Agilent 8453 UV/Vis spectrophotometer. ^1H NMR spectrum was recorded in CDCl_3-d_6 on a Varian UNITY INOVA 500 MHz spectrophotometer using TMS as internal reference. Mass spectra were performed on a Thermo Finnigan LCQ Advantage Max LC/MS/MS. Melting point was determined on Perkin Elmer model: TG/DTA 6300 under nitrogen atmosphere. Elemental analysis was performed on a Thermo Flash EA 1112.

2.2. Synthesis of (1E,2E)-3-[4-(Dimethylamino)phenyl]prop-2-en-1-ylidene)phenylamine (SBL). A solution of aniline (1) (0.79 g, 8.56 mmol) in 20 mL dry ethanol was added dropwise to a solution of (2E)-3-[4-(dimethylamino)phenyl]acrylaldehyde and (2) (1.50 g, 8.56 mmol) in 10 mL dry ethanol, and the mixture was stirred under argon for 12 h at 90°C . The solvent was evaporated to 1/2 of the initial volume, and petroleum ether was added at room temperature. A yellow precipitate was obtained when the solution was cooled to room temperature. It was filtered off and then recrystallized from 2:1 n-hexane/ethylacetate (Figure 1). Yield 81%, m.p. 138.8°C , FTIR $\nu_{\text{max}}/\text{cm}^{-1}$: 3033 (Ar, w), 2899–2809 ($-\text{CH}_3-$, m), 1662 ($\text{HC}=\text{N}$, s). ^1H NMR (CDCl_3-d_6) δ , ppm: 8.1 (d, H, $\text{HC}=\text{N}$) (disappeared with D_2O), 6.6–7.6 (m, 4H, Ar-H), 7.0 (m, 2H, $-\text{CH}=\text{CH}-$), 2.92 (s, 6H, N-(CH_3) $_2$). LC-MS (APCI) m/z (100%): 250.937 $[\text{M}]^+$. Anal. Calc. for $\text{C}_{17}\text{H}_{18}\text{N}_2$ (%): C, 81.56; H, 7.25; N, 11.19. Found (%): C, 81.59; H, 7.23; N, 11.18.

2.3. BET and SEM Analyses. BET analysis was carried out to determine the BET surface area and pore size of SBL. The surface area and pore size are very important application parameters as the surface contributes to the potential for interaction between the pigment and the polymer matrix [16]. Quantachrome Ins. Quadrasorb SI model BET was

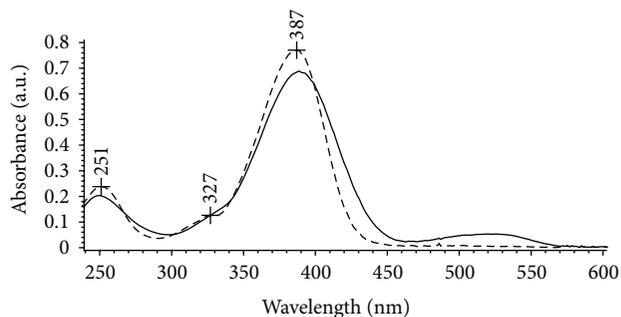


FIGURE 1: The UV-Vis spectra of (2E)-3-[4-(dimethylamino)phenyl]acrylaldehyde (line) and the Schiff base ligand (dashed line) in ethanol at room temperature.

used in this study. The measurements were carried out under nitrogen atmosphere. SEM analysis was carried out to determine the shape of SBL with Zeiss model: EVO LS 10.

2.4. Inverse Gas Chromatography (IGC) Measurements. A Hewlett-Packard 6890 Model, series II, gas chromatograph with a thermal conductivity detector was used to measure the retention time of the solvents in this study. The column was stainless steel tubing with 3.2 mm o.d. and 0.5 m in length. The SBL was coated on the support by slow evaporation of chloroform as stirring the Chromosorb W in the SBL solution. Trace amount of solvent was injected into the chromatograph. The column was conditioned at 110°C for 24 h under helium atmosphere. The probes were of high purity grade n-alkanes such as n-hexane (Hx), n-heptane (Hp), n-octane (O), n-nonane (N), and other acidic, basic, and amphoteric probes such as tetrahydrofuran (THF, basic), dichloromethane (DCM, acidic), chloroform (TCM, acidic), acetone (AC, amphoteric), and ethyl acetate (EA, amphoteric) were used without further purification. The all studied solvents and support materials being Chromosorb W (AW-DMCS-treated, 80/100 mesh) were supplied from Merck AG, Inc. Silane treated glass wool used to plug the ends of the column was obtained from Alltech Associates, Inc.

2.5. IGC Theory. The IGC technique provides a means of gaining information concerning the surface polarity of particulates and the acid-base nature of particulates. It provides a basis for determining the potential for chemical interaction between the pigment and its medium (polymer matrix or paint solutions, etc.). The material to be investigated is immobilised within a chromatographic column which is flushed through with inert gas such as helium or nitrogen. The stationary phase characterisation is achieved by utilising the partitioning of the sample between the mobile phase and the stationary phase, indicated by the time taken to elute the sample. The probe-probe interactions in IGC experiments are negligible because of being carried out in infinite dilution. The theory and the technique are now quite well described in the literature [16, 17].

The adsorbate net retention volumes V_N were calculated from the following expression:

$$V_N = \frac{Q * J * (t_R - t_A) * T}{(T_f)} \quad (1)$$

where t_R is the adsorbate retention time, t_A is the retention time of air, Q is volumetric flow rate measured at column outlet and at ambient temperature T_f (K), T is the column temperature (K), and J is the James-Martin gas compressibility correction factor [18].

The dispersive component of surface energy was determined using both the Dorris-Gray and the Fowkes methods. The adsorption energy for the n -alkanes increases with the number of carbon atoms in the chain. According to Dorris & Gray (1980), the increment, corresponding to the adsorption energy of a methylene group, $\Delta G_{A[CH_2]}$ is given by [19]

$$\Delta G_{A[CH_2]} = -RT \ln \left(\frac{V_{N,n}}{V_{N,n+1}} \right) \quad (2)$$

where $V_{N,n}$ and $V_{N,n+1}$ are the retention volumes of two n -alkanes having n and $n + 1$ carbon atoms in their chain. This parameter is independent of the chosen state of the adsorbed molecule. Thus, at constant temperature, for a series of alkane probes, a plot $RT \ln V_N$ versus the number of carbon atoms should give a straight line from which $\Delta G_{A[CH_2]}$ can be found.

The methylene adsorption energy can also be defined as [19]

$$\Delta G_{A[CH_2]} = 2N_A a_{[CH_2]} \sqrt{\gamma_S^D \gamma_{L[CH_2]}}, \quad (3)$$

where N_A is Avogadro's number, $a_{[CH_2]}$ is the surface area covered by one methylene group (0.06 nm^2), and $\gamma_{L[CH_2]}$ is the surface free energy of a surface consisting of methylene groups, that is, polyethylene, given by

$$\gamma_{L[CH_2]} = 35.6 + 0.058 (293 - T). \quad (4)$$

Thus, using (2)–(4) and the experimentally determined values of $V_{N,n}$ and $V_{N,n+1}$, the dispersion component of the surface free energy, γ_S^D may be calculated.

The free energy of adsorption ΔG_A may also be defined in terms of the retention volume of the probes [20]:

$$\Delta G_A = -RT \ln (V_N) + K. \quad (5)$$

T is the column temperature and K is a constant for a given column.

Consequently, the equations may be combined to give the Fowkes equation [21]:

$$-\Delta G_A = RT \ln (V_n) = 2Na(\gamma_S^D)^{0.5} (\gamma_L^D)^{0.5} + K''. \quad (6)$$

Thus, for a series of n -alkane probes, a plot of $RT \ln V_N$ against $a(\gamma_L^D)^{0.5}$ will give a slope of $2N(\gamma_S^D)^{0.5}$. Values of $a(\gamma_L^D)^{0.5}$ and boiling point T_b ($^\circ\text{C}$) of apolar solvents are found in the literature [22, 23].

The specific component of the free energy is determined from the n -alkane plot of $RT \ln(V_N)$ against $a(\gamma_L^D)^{0.5}$. The distance between the ordinate values of the polar probe datum point and the n -alkane reference line gives the specific component of the surface free energy, $-\Delta G_A^S$.

An equation may be written for this procedure,

$$-\Delta G_A^S = RT \ln \left(\frac{V_{N,n}}{V_{N,\text{ref}}} \right), \quad (7)$$

where $V_{N,n}$ and $V_{N,\text{ref}}$ are the retention volume for the polar probe and the retention volume for the n -alkanes' reference line, respectively.

The adsorption of a polar probe onto the adsorbent surface leads to a change in the enthalpy of the system and the entropy of the system. These factors are related to the energy of adsorption by the following:

$$\Delta G_A^S = \Delta H_A^S - T\Delta S_A^S. \quad (8)$$

Here, ΔH_A^S is the adsorption enthalpy by the Lewis acid-base interactions, ΔS_A^S is the adsorption entropy the Lewis acid-base interactions, and T is the column temperature. For each polar probe, ΔH_A^S and ΔS_A^S can be determined from a plot of $-\Delta G_A^S/T$ against $1/T$.

The surface Lewis acidity and basicity constants, K_A and K_D , may be calculated from the following,

$$-\Delta H_A^S = K_A DN + K_D AN^*. \quad (9)$$

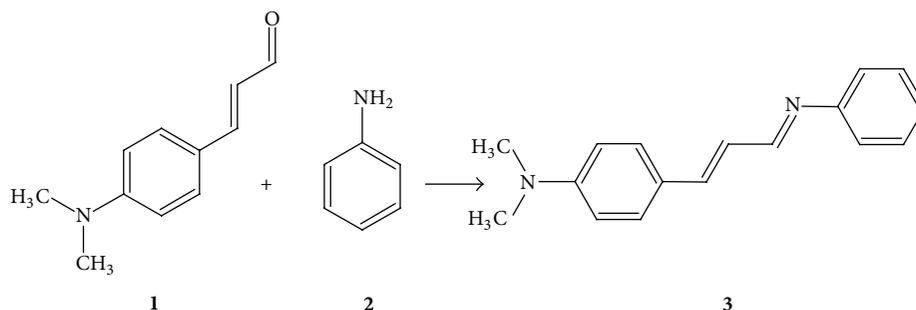
Here, DN and AN^* are Gutmann's donor and modified acceptor numbers, respectively. Values of $a(\gamma_L^D)^{0.5}$, boiling point, T_b ($^\circ\text{C}$), Gutmann's modified acceptor number, AN^* , and donor number, DN , of the polar probes used in this study are taken from [23, 24].

K_A and K_D are obtained from a plot of $-\Delta H_A^S/AN^*$ versus DN/AN^* with K_A as the slope and K_D as the intercept. Parameters K_A and K_D reflect the ability of the examined surface to act as an electron acceptor and electron donor, respectively [20].

3. Results and Discussion

The new Schiff base ligand was synthesized from the reaction of (2E)-3-[4-(dimethylamino) phenyl]acrylaldehyde with aniline, in a good yield, as shown in Scheme 1. The IR spectrum of the SBL shows the characteristic Schiff base stretching band at 1662 cm^{-1} . This intense band is assigned to the $-\text{C}=\text{N}$ stretching frequency of the ligand and is characterized for the azomethine moiety of most of the Schiff base compounds. The absorption band of the $\text{C}=\text{O}$ in (2E)-3-[4-(dimethylamino) phenyl]acrylaldehyde disappeared in the infrared spectrum of the ligands, which indicates that the condensation has occurred. The alkyl and aryl bands of the ligand are observed at 2809–2899, 1364–1448, and 1149–1151 cm^{-1} , respectively.

Elemental analysis of the Schiff base ligand shows good agreement with the proposed structures. The ^1H NMR



SCHEME 1: Synthesis of the Schiff base ligand (SBL).

spectrum of the SBL in CDCl_3-d_6 confirmed the proposed structure showing one D_2O -exchangeable proton at 8.1 ppm for the $\text{HC}=\text{N}$ group. The assignments of the protons are highly complicated in the regions of 2.92 and 6.6–7.6 ppm, where the signals are due to the protons of the alkyl and aromatic groups.

The SBL is stable at room temperature but hygroscopic. The SBL is soluble in common polar organic solvents, such as ethanol, methanol, and chloroform but partially soluble in nonpolar organic solvents, such as benzene and hexane.

The UV-Vis spectral data for the SBL ligand show low-energy band at approximately 249 nm and high energy band at approximately 389 nm, due to $n-\pi^*$ and $\pi-\pi^*$ transitions of the azomethine group in the ligands [25, 26] (Figure 1).

According to UV spectra, the peaks recorded in the spectrum are broadened and slightly red shifted (Figure 1).

In the mass spectra, the molecular ion peak of the L appeared at m/z (100%): 250.937 $[\text{M}]^+$. Mass spectral data confirmed the proposed structure of the Schiff base ligand (Figure 2).

According to BET results, the surface area and pore size of SBL were determined as $32.4 \text{ m}^2 \cdot \text{g}^{-1}$ and 3.056 nm, respectively. This ligand has a potential to be used in nanotechnology paint applications. The SEM micrograph of SBL was given in Figure 3.

The shape of SBL was determined as a rod-like structure from SEM micrograph. The retention diagrams of nonpolar and polar solvents on SBL were plotted net retention volumes, V_N , of solvents on SBL which were calculated from IGC measurements between 30°C and 60°C by using (1) versus inverse temperature. According to (2), $\Delta G_{A[\text{CH}_2]}$ is independent of the chosen reference state of adsorbed molecule. The $RT \ln V_N$ versus carbon number of n -alkanes were plotted in Figure 4.

The slope of the fitted line is equal to $\Delta G_{A[\text{CH}_2]}$. The variation of γ_S^D according to the Dorris-Gray approach and $\gamma_{L[\text{CH}_2]}$ with temperature were calculated from (3) and (4), respectively. According to Fowkes, calculated values of $RT \ln V_N$ were plotted against $a(\gamma_L^D)^{0.5}$. An example of the pattern of results obtained was given in Figure 5 for the isotherm at 303 K.

The results of $\Delta G_{A[\text{CH}_2]}$, $\gamma_{L[\text{CH}_2]}$, and γ_S^D according to the Dorris-Gray and the Fowkes approaches were given in Table 1.

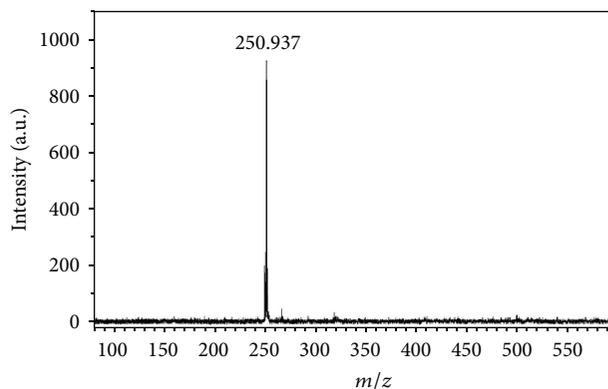


FIGURE 2: Mass spectrum of SBL.

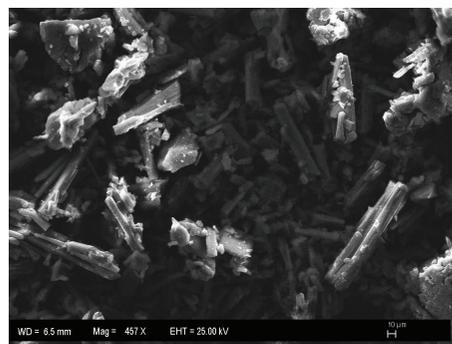


FIGURE 3: The SEM micrograph of SBL.

The values of γ_S^D calculated according to the Dorris-Gray and the Fowkes approaches are very close each to other at the studied temperature ranges. Compared to the values with the before published other Schiff base ligand γ_S^D values, the γ_S^D of (*E*)-*N*-((*E*)-3-(4-(dimethylamino)phenyl)allylidene)-4-(4-((*E*)-((*E*)-3-(4(dimethylamino)phenyl)allylidene) amino)-3-methylnaphthalen-1-yl)-2-ethylnaphthalen-1-amine as determined as $33.93 \text{ (mJ/m}^2\text{)}$ at 303 K according to the Dorris-Gray approach [7]. The values are different due to chemical nature difference between the two ligands. It can be said that when the phenyl ring is added to the Schiff base structure, γ_S^D of ligand is increasing.

TABLE 1: The adsorption energy of a methylene group, $\Delta G_{A[CH_2]}$, the surface free energy of a surface consisting of methylene groups, $\gamma_{L[CH_2]}$ and dispersion component of surface free energy, γ_S^D , values calculated by the Dorris-Gray and the Fowkes approaches for L determined at studied temperatures.

| T (K) | $\gamma_{L[CH_2]}$ (mJ/m ²) | $\Delta G_{A[CH_2]}$ (10 ⁶ mJ/mol) | Dorris-Gray γ_S^D (mJ/m ²) | Slope ($\times 10^{24}$) | Fowkes γ_S^D (mJ/m ²) |
|---------|---|---|---|----------------------------|--|
| 303 | 35.02 | 2.25 | 27.80 | 6.33 | 27.65 |
| 313 | 34.44 | 2.12 | 24.95 | 6.17 | 26.23 |
| 323 | 33.86 | 2.02 | 23.19 | 6.06 | 25.36 |
| 333 | 33.28 | 1.87 | 20.09 | 5.78 | 23.08 |

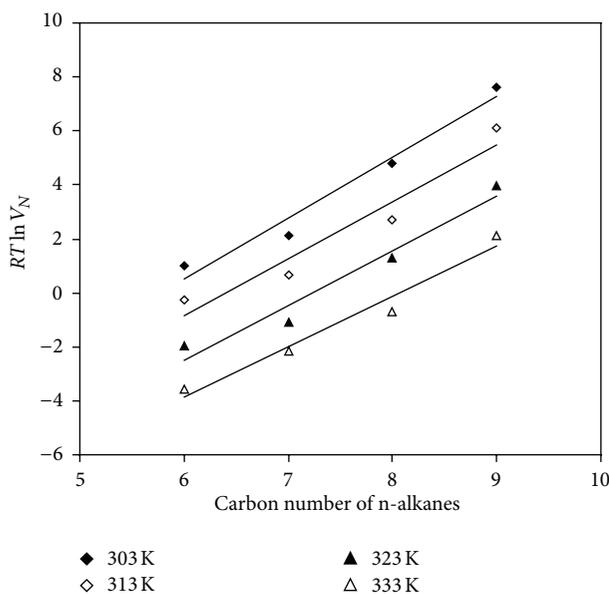


FIGURE 4: The plot of $RT \ln V_N$ versus carbon number of n -alkanes.

The specific component of the surface free energy, $-\Delta G_A^S$, is calculated using the difference between the calculated value of $RT \ln V_N$ and that which was derived using the equation of the linear fit of the n -alkane reference line. By plotting the values of $-\Delta G_A^S/T$ against $1/T$, the adsorption enthalpy, ΔH_A^S , and the adsorption entropy, ΔS_A^S were determined for each studied polar probe. The values of K_A and K_D were calculated using (9). The plotting $-\Delta H_A^S/AN^*$ versus DN/AN^* with K_A as the slope and K_D as the intercept (Figure 6).

The values of K_A and K_D are found to be 0.1 and 0.6, respectively. According to the values obtained for K_A and K_D , the surface of SBL is the basic character between 30–60°C.

4. Conclusions

In this study, a new Schiff base ligand with D- π -D type has been successfully synthesized using the reaction of (2E)-3-[4-(dimethylamino) phenyl]acrylaldehyde with aniline and characterized by FTIR, UV-visible, ¹H NMR, elemental analysis, and mass spectrum for structural characterization and BET, SEM, and IGC for surface characterization. The big surface area and small pore size of SBL were determined

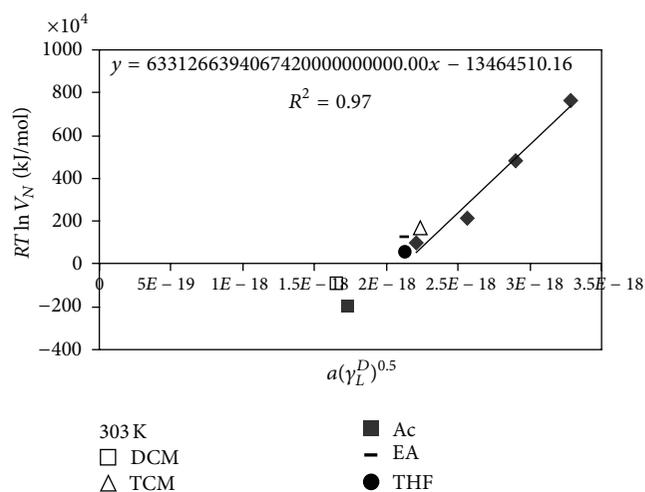


FIGURE 5: A plot of $RT \ln V_N$ versus $a(\gamma_L^D)^{0.5}$ for n -alkanes and polar probes on SBL at 303 K.

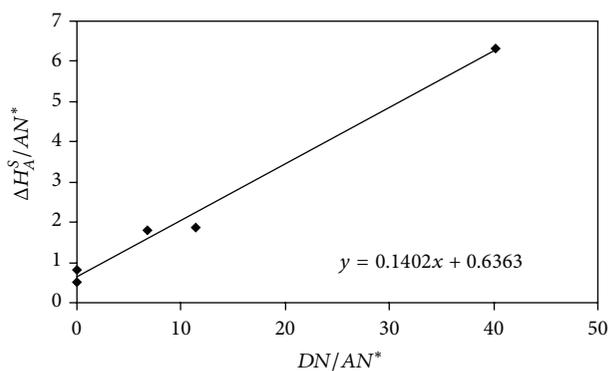


FIGURE 6: The plot of $-\Delta H_A^S/AN^*$ versus DN/AN^* .

from the BET result. It can be used in nanotechnology paint applications. In addition, IGC was applied to the investigation of adsorption properties of the SBL. The relation of SBL with used polar and nonpolar solvents was tested at temperatures between 30°C and 60°C in which SBL does not show any thermal transition. The base constant of newly synthesized SBL was determined as 0.64, while acid constant is 0.1.

Conflict of Interests

All the authors declare no financial relationships with Merck or any companies.

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