

GUSTINE I VISKOZNOSTI BINARNIH SISTEMA SA N,N-DIMETILANILINOM I TETRA ETILEN GLIKOLOM DIMETIL ETROM ILI N-METIL-2-PYROLIDONOM, KAO POTENCIJALNI RASTVARAČI ZA PROCESSE ODSUMPORAVANJA DIMNIH GASOVA

DENSITIES AND VISCOSITIES OF BINARY SYSTEMS WITH N,N-DIMETHYLANILINE AND TETRA ETHYLENE GLYCOL DIMETHYL ETHER OR N-METHYL-2-PYRROLIDONE, AS POTENTIAL SOLVENTS FOR FLUE GAS DESULPHURIZATION PROCESSES

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Oksidi sumpora emituju se kao jedan od proizvoda u procesu sagorevanja čvrstih fosilnih goriva u termoelektranama, posebno kolubarskog i kostolačkog lignita u Republici Srbiji. Da bi se smanjio nivo emisije indirektnih gasova staklene bašte (GHG) iz stacionarnih izvora, neizbežna je primena tehnologije odsumporavanja dimnih gasova (FGD). Pored klasičnog krečnjačkog postupka, sve su češći tehnološki postupci zasnovani na fizičkoj ili hemijskoj apsorpciji sumpornih oksida u rastvaraču i njegovoj termičkoj regeneraciji. Da bi se pravilno dizajnirao i optimizovao proces, važno je precizno odrediti termofizička svojstva čistih komponenti i njihovih smeša.

U radu su prikazani rezultati određivanja gustine i viskoziteta dve binarne smeše, N,N-dimetilanilin (N,N-DMA) + tetra etilen glikol dimetil etar (TEGDME) i N,N-dimetilanilin (N,N-DMA) + N-metil-2-pirolidon (NMP). Merenja su vršena pomoću gustinomera Anton Paar DMA 5000 i Stabinger viskozimetra Anton Paar SVM 3000/G2, na atmosferskom pritisku. Dobijeni eksperimentalni rezultati su prikazani kao funkcija temperature i molskog udela N,N-DMA.

N,N-DMA pripada grupi amina, a korišćen je kao sorbent za prečišćavanje dimnih gasova u procesima sa termičkom regeneracijom (CANSOLV, Sulphidine i ASARCO procesi). Druga dva organska rastvarača su TEGDME i NMP. Posebna pažnja je posvećena NMP-u zbog niske cene i niskog viskoziteta, sa svojim povoljnim svojstvima, što ga čini konkurentnim kao rastvarač za FGD. TEGDME je polarni rastvarač, ali nije selektivan ka SO₂ i druge gasovite komponente, pa je NMP predložen kao alternativno rešenje. Regenerativni proces za uklanjanje SO₂, sa TEGDME, već je našao komercijalnu primenu (Solinox proces). NMP se već industrijski koristi kao rastvarač u Lurgi's Purisol procesu, zbog svoje prirode za selektivno odsumporavanje dimnih gasova.

Ključne reči: gusina; viskoznost; N,N-DMA; TEGDME; NMP; odsumporavanje

Sulfur oxides are emitted as one of the products in the process of combusting solid fossil fuels in thermal power plants, especially the Kolubara and Kostolac lignites in the Republic of Serbia. In

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order to reduce the emission level of the indirect Greenhouse gases (GHG) from stationary sources, it is inevitable to implement flue gas desulfurization (FGD) technology. In addition to the classic limestone process, technological procedures based on the physical or chemical absorption of sulfur oxides in the solvent and its thermal regeneration are becoming increasingly common. In order to properly design and optimize the process, it is significant to precisely determine the thermophysical properties of pure components and their mixtures.

The paper presents the results of determining the density and viscosity of two binary mixtures, *N,N*-Dimethylaniline (*N,N*-DMA) + Tetra Ethylene Glycol Dimethyl Ether (TEGDME) and *N,N*-Dimethylaniline (*N,N*-DMA) + *N*-methyl-2-pyrrolidone (NMP). Measurements were made using a densitometer Anton Paar DMA 5000 and Stabinger viscometer Anton Paar SVM 3000/G2, at atmospheric pressure. The obtained experimental results are presented as a function of temperature and *N,N*-DMA mole fraction.

N,N-DMA belongs to the group of amines, and it has been used as a sorbent for purification of flue gases in processes with thermal regeneration (CANSOLV, Sulphidine and ASARCO processes). The other two organic solvents are TEGDME and NMP. Special attention was paid to NMP due to its low cost and low viscosity, with its favorable properties, making it competitive as a solvent for FGD. TEGDME is a polar solvent, but it is not selective to SO₂ and other gaseous components, so NMP has been suggested as an alternative solution. A regenerative process for SO₂ removal, with TEGDME, has already found commercial application (Solinox process). NMP is already industrially used as a solvent in Lurgi's Purisol process, due to its nature for selective desulfurization of flue gases.

Keywords: density; viscosity; *N,N*-DMA; TEGDME; NMP; desulfurization.

1. Introduction

Sulfur dioxide (SO₂) is toxic, irritating compound with unpleasant odour, which is in gaseous state at atmospheric pressure. At normal conditions, it is soluble in water to form sulfurous acid (H₂SO₃). During oxidation process, sulfur trioxide (SO₃) is formed. Dissolved SO₃ in water forms sulfuric acid (H₂SO₄). The affinity of sulfur oxides (SO_x) to form acids with water or air moisture, puts them as undesirable constituents of the atmosphere. As an environmental pollutant SO₂ has multiple harmful effects which are already well known. Industrial activities that involve a combustion of fossil fuels that contain sulfur, such as coal and oil in power plants, foundries and steel mills, have significant impact.

The most common method for FGD is the lime/limestone process, which was patented in 1909 [1], and since then has gained wide practical application. Even today, the lime/limestone process, with some improvements/modifications [2-5], is the major FGD method, especially on large thermal power and industrial plants. The alkaline reagent for lime/limestone wet non-regenerative procedure is a mixture of calcium oxide (CaO), calcium hydroxide (Ca(OH)₂) or calcium carbonate (CaCO₃) with water. The final product of this process is commercially useful product - gypsum.

In recent years, regenerative processes with the physical absorption in liquid organic solvents followed by solvent thermal regeneration, are getting more importance and consequently more frequent application [1, 6-8]. The procedure becomes more significant, because of its advantages over other methods: the end product has its market value, the accumulation of large amounts of by-products in the solid state is avoided (drawback of the lime / limestone process), low investment and operating costs and high flexibility in terms of process parameters, size of the plant, flow of flue gases, SO₂ concentration etc. In order to avoid major losses from the process, the selection of the

organic solvent is of key importance. Physical absorption process is not energy demanding one, considering that the binding energy due to physical interactions between solvent molecules and SO₂ is lower than the one in the processes of chemical absorption which include chemical reactions and the formation of new compounds.

This paper presents novel data on density and viscosity of N,N-DMA with TEGDME and N,N-DMA with NMP, binary mixtures in the entire composition range, at temperatures between 288.15 K and 323.15 K with 5 K temperature step, at pressure 0.1 MPa.

N,N-DMA, as tertiary amine, has high selectivity towards SO₂ in comparison to the other components of flue gas like CO₂, and excellent binding capability by the mechanisms of chemical absorption. The main disadvantage of N,N-DMA is its high toxicity which imposes the need for more environmentally friendly replacements, or mixing with them. It has been used as solvent in *CANSOLV*, *Sulphidine* and *ASARCO* processes [8].

Tetraethylene glycol dimethyl ether is a polar solvent that is thermally and chemically stable, with a high boiling point, non-toxic but has an irritating effect. TEGDME has found practical application in regenerative processes for SO₂ removal from flue gases [9, 10], however, it is not selective for sulfur dioxide, in relation to other gaseous components. NMP also has very desirable properties, such as low volatility (high boiling point), low flammability and relatively low toxicity. In contrast to TEGDME, in terms of selectivity towards SO₂, NMP meets all the affirmative properties for the solvent preferred for separation processes.

2. Materials and methods

The chemicals with purity guaranteed by the manufacturer that were used for the experiments are: N,N-dimethylaniline (Merck, ≥ 99.0%), TEGDME (Merck, ≥ 98.0%) and NMP (Merck, ≥ 99.0%). Before use, the chemicals were kept in a dry, dark place in the original bottles. The chemicals were used without prior purification and degassed before use. Comparison of values of properties obtained by experiments of pure chemicals with available data from the literature, at atmospheric pressure and temperatures of 298.15 and 303.15 K, for density is given in Table 1, and for dynamic viscosity in Table 2.

Density measurements were performed on an Anton Paar DMA 5000 instrument, while viscosities of pure chemicals and binary mixtures were measured with a digital Anton Paar Stabinger viscometer (model SVM 3000/G2) with an accuracy of ± 0.1% of the measured value. The temperature in the cell is regulated by a thermostat located in the instrument with an uncertainty of ± 0.01 K. A detailed description of the apparatus and the measurement procedure is given in our previous works [11, 12, 13]. The mixtures were prepared by measuring the mass of pure substances and mixtures on a Mettler AG 204 balance with a precision of 1×10⁻⁷ kg. The standard uncertainty of mole fraction values is ± 1×10⁻⁴, while the extended relative uncertainty (k ≈ 2) of viscosity measurement is ± 0.9%.

When mixing two or more miscible components, a change in mixture volume occurs, compared to the ideal one. Excess molar volume (V^E) represents a quantitative change in volume during mixing, considering the following factors [24, 25]:

- differences in the size and shape of the molecules of the components,
- formation of new chemical bonds and
- structural changes.

The excess molar volume (V^E) of a mixture is defined as follows:

$$V^E = V - \sum_{i=1}^N x_i V_i \quad (1)$$

where: V – molar volume of the mixture, V_i – molar volume of pure component i , x_i – mole fraction of component i , N – number of components of the mixture.

Table 1. Densities (ρ) of pure components at $T = 298.15$ and $T = 303.15$ K at $p = 0.1$ MPa

Component	T [K]	ρ [g·cm ⁻³]	
		Exp.	Lit.
N,N-DMA (Merck)	303.15	0.947778	0.9484 [14] 0.948 [15] 0.94833 [16]
TEGDME (Merck)	298.15	1.006263	1.0063 [17] 1.0067 [18] 1.0059 [19] 1.00653 [20]
NMP (Merck)	298.15	1.028382	1.02872 [21] 1.0283 [22] 1.02831 [23]

Table 2. Dynamic Viscosities (η) of pure components at $T = 298.15$ and $T = 303.15$ K at $p = 0.1$ MPa

Component	T [K]	η [mPa·s]	
		Exp.	Lit.
N,N-DMA (Merck)	303.15	1.2035	1.174 [14] 1.170 [15]
TEGDME (Merck)	298.15	3.3389	3.394 [17] 3.313 [19]
NMP (Merck)	298.15	1.6795	1.656 [21] 1.67 [22] 1.663 [23]

For binary mixtures, expression (1) takes the form (2):

$$V^E = \frac{x_1 M_1 + x_2 M_2}{\rho} - \left(\frac{x_1 M_1}{\rho_1} + \frac{x_2 M_2}{\rho_2} \right) \quad (2)$$

where: x_1 and x_2 – mole fractions of components, M_1 and M_2 – molar masses of pure components, ρ_1 and ρ_2 – densities of pure components and ρ – density of the binary mixture.

The proportionality coefficient between the tangential stress and the velocity gradient is called the dynamic viscosity η (Pas), and figures in the expression for the tangential stress, [25]:

$$\tau_{xy} = \eta \cdot \left(\frac{dv_x}{dy} \right) \quad (3)$$

where τ_{xy} - tangential stress, (dv_x/dy) – velocity gradient and η - dynamic viscosity.

The viscosity of the liquid does not depend significantly on the pressure, and as the temperature increases, the viscosity decreases [26].

As in the case of the excess volume, when mixing two, three or more miscible components, there is a deviation in dynamic viscosity ($\Delta\eta$) in relation to the viscosity of the ideal mixture. The dynamic viscosity deviation is given by the expression:

$$\Delta\eta = \eta - \sum_{i=1}^N x_i \eta_i \quad (4)$$

where: η - viscosity of the binary mixture, η_i - the viscosity of the pure component i and x_i - mole fraction of component i .

Deviation in dynamic viscosity during the formation of binary mixtures is determined based on experimentally determined dynamic viscosities of the mixture and components, and is calculated according to (5):

$$\Delta\eta = \eta - (x_1\eta_1 + x_2\eta_2) \quad (5)$$

where x_1 and x_2 represent mole fractions of components, η_1 and η_2 - viscosities of pure components and η , viscosity of the binary mixture.

Experimental data of binary systems is correlated by the *Redlich-Kister* polynomial expression [27]:

$$Y_{ij} = x_i x_j \sum_{k=1}^m A_k (x_i - x_j)^{k-1} = x_i x_j \sum_{k=1}^m A_k (2 \cdot x_i - 1)^{k-1} \quad (6)$$

where: A_k - polynomial parameters, i, j - components of the binary system and Y_{ij} - excess molar volume (V^E) or deviation in viscosity ($\Delta\eta$) of the binary system.

3. Results and discussion

The binary system N,N-DMA+TEGDME consists of two mutually soluble polar molecules, weak intermolecular interactions are present, with the shape of the "S" curve, Figure 1. For mole fractions of N,N-DMA in the range $x_1=0\div 0.6$, positive V^E values were registered as a consequence of poor spatial "packing" of different molecules and mutually repulsive dipole-dipole interactions of methyl groups ($-CH_3$) present in both molecules. For larger mole fractions of N,N-DMA $x_1=0.6\div 1.0$, the expansion and breaking of hydrogen bonds between oxygen and hydrogen atoms and "straightening" of the TEGDME chain molecule occurs, which enables a better mutual spatial distribution of the components.

In contrast to the N,N-DMA+TEGDME system, strong attractive intermolecular interactions are present in the N,N-DMA+NMP system, Figure 2. The negative values of the excess molar volumes are a consequence of the high polarity of the NMP molecule. A significant contribution to the strong attractive interactions is made by hydrogen bonds formed between nitrogen atoms (NMP and N,N-DMA) and oxygen atoms (NMP) and hydrogen atoms, molecules of the first and second components. A negligible influence of temperature on V^E values is noticeable, Figure 2.

Figure 3. presents the deviation of dynamic viscosity, in the temperature range 288.15 K \div 323.15 K. The dynamic viscosity deviation values for the N,N-DMA+TEGDME system, at all compositions and temperatures, are positive. The maximum value of dynamic viscosity deviation of the system is at the composition $x_1\approx 0.5$, which is about 0.1 Pas.

Due to the attractive intermolecular interactions, in the binary system N,N-DMA+NMP, there is a better and more compact "packing" of molecules and a reduction in volume. Denser packing and volume reduction, on the other hand, cause a positive deviation in dynamic viscosity shown in Figure 4. The minimum value of the excess molar volume has been obtained at the fraction $x_1\approx 0.5$. With the same composition of the binary mixture, the maximum value of the dynamic viscosity deviation is about 0.16 Pas which is achieved at a temperature of 288.15 K.

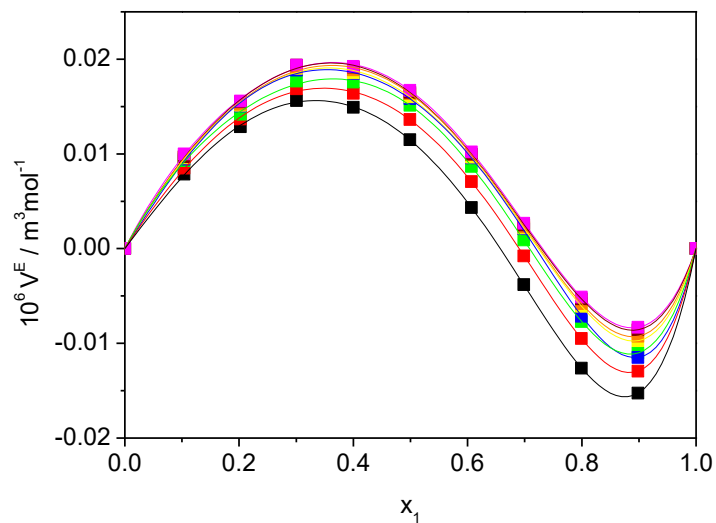


Figure 1. Experimental values of excess molar volumes for the systems: *N,N-DMA+TEGDME*, depending on the mole fraction of *N,N-DMA*, in the temperature interval from 288.15 K to 323.15 K,
 ■ – 288,15 K, ■ – 293,15 K, ■ – 298,15 K, ■ – 303,15 K,
 ■ – 308,15 K, ■ – 313,15 K, ■ – 318,15 K, ■ – 323,15 K.

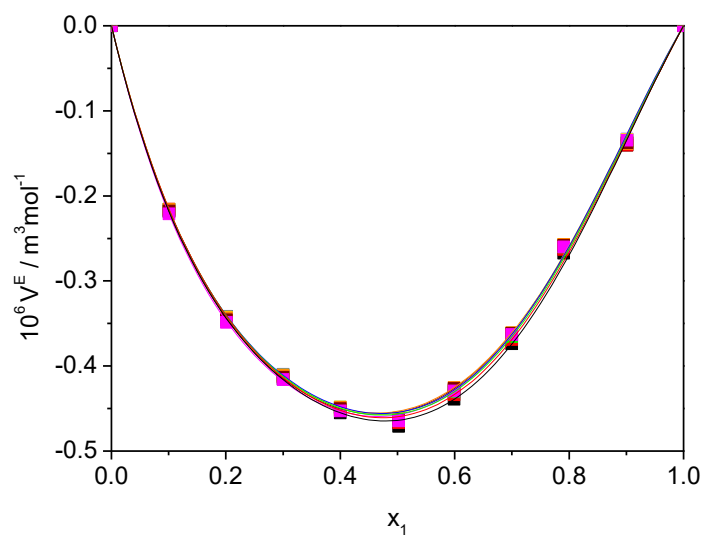


Figure 2. Experimental values of excess molar volumes for the systems: *N,N-DMA+NMP*, depending on the mole fraction of *N,N-DMA*, in the temperature interval from 288.15 K to 323.15 K,
 ■ – 288,15 K, ■ – 293,15 K, ■ – 298,15 K, ■ – 303,15 K,
 ■ – 308,15 K, ■ – 313,15 K, ■ – 318,15 K, ■ – 323,15 K.

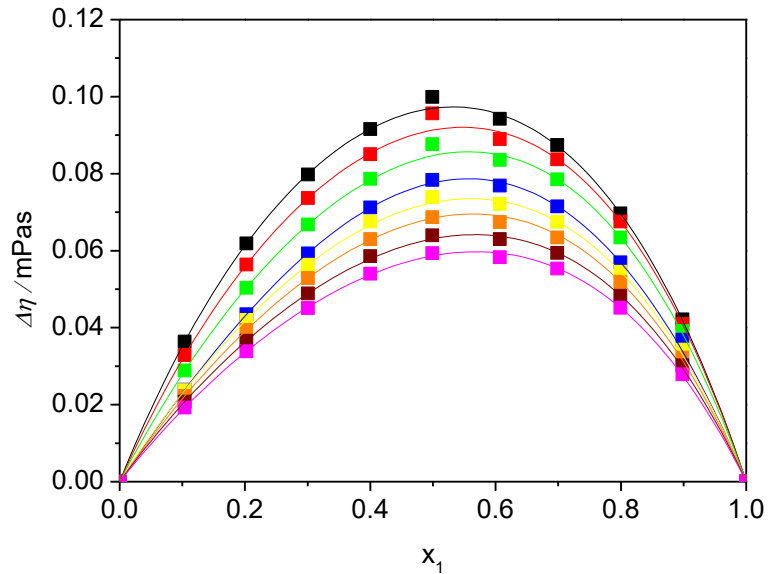


Figure 3. Experimental values of deviation dynamic viscosity for *N,N*-DMA+TEGDME, depending on the mole fraction of *N,N*-DMA, in the temperature interval from 288.15 K to 323.15 K, ■ – 288,15 K, ■ – 293,15 K, ■ – 298,15 K, ■ – 303,15 K, ■ – 308,15 K, ■ – 313,15 K, ■ – 318,15 K, ■ – 323,15 K.

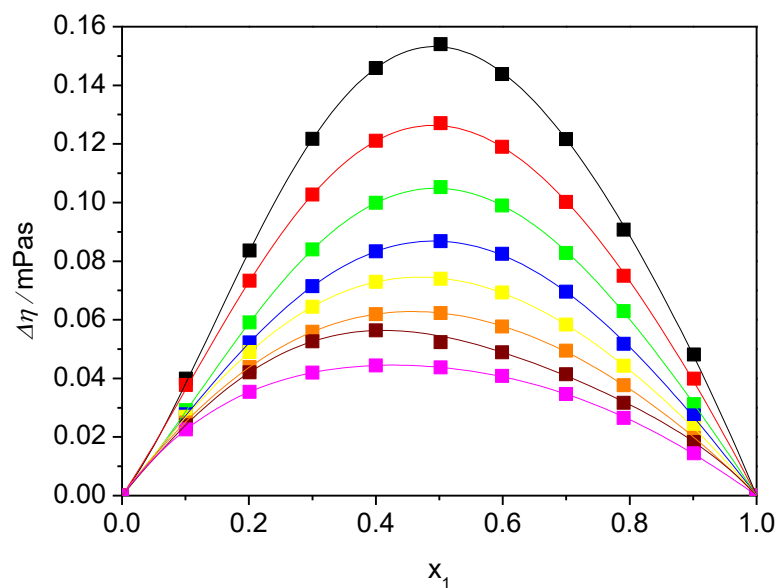


Figure 4. Experimental values of deviation of dynamic viscosity for systems DMA+NMP, depending on the mole fraction of *N,N*-DMA, in the temperature interval from 288.15 K to 323.15 K, ■ – 288,15 K, ■ – 293,15 K, ■ – 298,15 K, ■ – 303,15 K, ■ – 308,15 K, ■ – 313,15 K, ■ – 318,15 K, ■ – 323,15 K.

4. Conclusion

The research of thermophysical and transport properties of two binary mixtures was conducted in this work. Chemicals and mixtures were selected on the basis of their potential for flue gas desulfurization processes. Measurements of density and viscosity, excess molar volumes and deviations in

viscosity have been calculated for all investigated mixtures and correlated with Redlich-Kister polynomial equation. The values of excess molar volumes and viscosity deviations were commented in relation to molecular interactions of existing binary mixtures.

For binary system N,N-DMA+TEGDME, very weak intermolecular interactions are present, as shown in Figure 1. For mole fractions $x_1=0\div 0.6$, positive V^E values exist due to poor spatial packing and mutual repulsive dipole-dipole interactions of methyl groups ($-CH_3$). For mole fractions $x_1=0.6\div 1.0$, better mutual spatial distribution of components prevails.

For N,N-DMA+NMP system, there are strong attractive intermolecular interactions, shown in Figure 2. The negative values of the excess molar volume are a consequence of the high polarity of the NMP molecules. A negligible influence of temperature on V^E values is noticeable. Denser packing and volume reduction, on the other hand, correspond to a positive change in dynamic viscosity.

5. Nomenclature

$V (m^3/mol)$	-	Molar volume
$V^E (m^3/mol)$	-	Excess molar volume
$V_i (m^3/mol)$	-	Molar volume of the component 'i'
x	-	Mole fraction
x_i	-	Mole fraction of the component 'i'
$M(kg/kmol)$	-	Molar mass
Y_{ij}	-	Excess properties, deviation of molar volume or dynamic viscosity of binary system
A_k	-	Parameters of Redlich – Kister polynomial
N	-	Number of experimental data
$v (m/s)$	-	Speed of fluid

Greek letters

$\rho(\frac{kg}{m^3})$	-	Density of binary mixture
$\rho_i(\frac{kg}{m^3})$	-	Density of component 'i'
$\eta (Pas)$	-	Dynamic viscosity of binary mixture
$\Delta\eta$	-	Deviation of dynamic viscosity
$\tau (Pa)$	-	Tangential stress

Subscripts

i, j	-	Indexes of pure components of binary system
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Superscripts

E	-	Excess property
N	-	Number of mixture components

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