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STUDY ON THE VISCOSITY OF SOLVENTS BASED ON ALANINE AND POTASSIUM CARBONATE

BY

RAMONA-ELENA TĂTARU-FĂRMUȘ¹, NICOLAE APOSTOLESCU¹,
CLAUDIA COBZARU¹, GABRIELA ANTOANETA APOSTOLESCU¹,
CORINA CERNĂTESCU¹, ELENA NICULINA DRĂGOI^{1*},
MIHAELA AURELIA VIZITIU¹ and MIHAELA POROCH²

¹“Gheorghe Asachi” Technical University of Iași, “Cristofor Simionescu” Faculty of Chemical Engineering and Environmental Protection, Iași, Romania

²“Grigore T. Popa” University of Medicine and Pharmacy Iași, Faculty of Medicine, Department of Preventive Medicine and Interdisciplinarity, Iași, Romania

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Abstract. Due to its low regeneration energy, low degradation rates, and low corrosivity, potassium carbonate is an excellent solvent for carbon dioxide capture. However, potassium carbonate has a drawback in that it has relatively slow reaction rate with CO₂, necessitating the use of large absorption apparatus. The addition of promoters to the potassium carbonate solutions is the most effective way to increase the absorption kinetics. Potassium salt of alanine (KAla) was chosen as a promoter for CO₂ absorption in potassium carbonate solution (25% K₂CO₃). The density and viscosity of solvents are crucial for their thorough evaluation for industrial applications, and they should be considered in addition to the absorption rate data. The viscosity of these novel solvents was evaluated over the temperature range of 298 - 333 K, for concentrations ranging from 3% KAla to the maximum 7% KAla.

Keywords: amino acid salts, carbon dioxide capture, chemical absorption, promoters, transport properties.

*Corresponding author; *e-mail*: elena.dragoi@tuiasi.ro

1. Introduction

Nowadays, the disastrous consequences of climate change determine a special attention on the energy generation process. Most of the energy is obtained from the burning of fossil fuels, a process that also determines greenhouse gas emissions, such as carbon dioxide (IPCC, 2007; IEA, 2020; Mac Dowell *et al.*, 2017). Therefore, current research is oriented towards the design and application of effective and low-cost CO₂ capture and separation technologies (Rubin *et al.*, 2012; Sang Sefidi and Luis, 2019).

Among the technologies currently used for carbon dioxide capture (post-combustion, pre-combustion, oxy-combustion), post-combustion CO₂ capture is the most frequently used (Mukherjee *et al.*, 2019; Godin *et al.*, 2021; Kanniche *et al.*, 2010; Raganati *et al.*, 2021). The basic difference between these post-combustion and pre-combustion CO₂ capture technologies is in the carbon dioxide separation step. In post-combustion technology, CO₂ is removed from the flue gas production after burning the fuel gas with air. In pre-combustion technology, CO₂ is first removed and then the remaining gas is burned with hydrogen. The fuel gas is burned with pure oxygen instead of air in the oxy-combustion process, which is a modified form of post-combustion technology (Mondal *et al.*, 2012; Rahman *et al.*, 2017).

As methods used for capturing carbon dioxide in the post-combustion stage, there are: chemical absorption, physical absorption, membrane separation, adsorption and cryogenic separation (Mukherjee *et al.*, 2019; Figueroa *et al.*, 2008). Among these, the most studied technology is chemical absorption, using various solvents: hot potassium carbonate solutions - promoted or unpromoted (Ayithey *et al.*, 2020; Hu *et al.*, 2016; Hu *et al.*, 2017; Ramezani *et al.*, 2018; Ramezani and Di Felice, 2021), amine solutions (Fan *et al.*, 2016; Hu *et al.*, 2017a; Nouroddinvand and Heidari, 2021; Tataru-Farmus *et al.*, 2007), ammonia solutions (Jilvero *et al.*, 2014; Kittiampon *et al.*, 2017; Olajire, 2013; Shakerian *et al.*, 2015; Zhao *et al.*, 2012), ionic liquids (Farsi and Soroush, 2020; Hospital-Benito *et al.*, 2020; Ramdin *et al.*, 2012), mixture of solvents (Ansaloni *et al.*, 2019; Eskandari *et al.*, 2022; Ochedi *et al.*, 2020; Rivera-Tinoco *et al.*, 2010; Wu *et al.*, 2020; Xiao *et al.*, 2019), etc. Industrially, absorption processes in potassium carbonate solutions, respectively absorption in amine solutions, have the greatest spread. A major challenge for CO₂ absorption systems with potassium carbonate solutions is their slow reaction kinetics compared to amine solution absorption systems, especially under post-combustion conditions (Harja *et al.*, 2021). Amines have some drawbacks, including: a significant energy loss for solvent regeneration, a high rate of deterioration, and corrosivity in the presence of oxygen (Harja *et al.*, 2020; Godin *et al.*, 2021).

Since several amino acid salts (AAS) solutions offer excellent qualities like a quick reaction rate, high CO₂ solubility, commercial availability, high

surface tension, low toxicity, and low volatility (Hu *et al.*, 2018; Sang Sefidi and Luis, 2019; Ramezani *et al.*, 2022; Tataru–Farmus *et al.*, 2022; Shen *et al.*, 2016), they are another class of solvents for CO₂ capture. They do, however, have some limitations, such as a large energy penalty for regeneration as a result of the production of permanent carbamates. As a result, there are studies into the use of these amino acid salts as a promoter in potassium carbonate solutions (Ahmed and Wiheeb, 2020; Hu *et al.*, 2017b; Li *et al.*, 2019).

For the full evaluation of solvents for industrial applications as well as for the modelling and design of gas-liquid contactors in acid gas treatment units, physico-chemical properties of solvents are required. The physical solubility, mass transfer, and dynamics of CO₂ in solvents are all dependent on density. When estimating the diffusivity with the modified Stoke-Einstein equation, viscosity is required, in order to calculate kinetic and mass transfer properties.

The objective of this paper is to present experimental data for viscosities of a new solvent, amino acid salts and potassium carbonate based and to represent the experimental results using statistical approaches as a function of concentration of amino acid salt (AAS) added and temperature.

2. Experimental

Materials

The chemicals were obtained as follows: DL-alanine (CAS 302-72-7) from GmbH, purity $\geq 99\%$, potassium carbonate (CAS 584-08-7), purity min. 99 % and potassium hydroxide (CAS 1310-58-3) purity min. 90% from ChimReactiv SRL, hydrochloric acid 37% HCl (CAS 7647-01-0) from BIOCHEM – Chemopharma. All reagents were used without further purification. Distilled water was used to prepare the solutions. Potassium carbonate (K₂CO₃) was weighed to prepare a solution equivalent to 25% K₂CO₃. The amino acid salt (AAS) - potassium salt of Alanine (KAla) - was prepared by neutralizing equimolar amounts of Alanine with KOH, according to reports in the literature (Sang Sefidi and Luis, 2019; Wei *et al.*, 2014; Eskandari *et al.*, 2022). The required amounts of KOH and Ala were calculated and weighed for a concentration of 3%, 5% and 7% KAla, respectively. First, K₂CO₃ was dissolved, and in the solution thus obtained, the amino acid was deprotonated; precipitates were not observed during this process. Since the reaction is exothermic, the mixing of the chemicals was carried out with cooling to avoid evaporation of water from the solution.

The amount of water formed, along with the water content of the potassium hydroxide, was considered when calculating the final concentration of the solutions. Using the titration method and the Phenolphthalein indicator, the specific concentration of each of the produced solvents was ascertained.

Method

Determination of viscosity

Viscosity was measured using an Ubbelohde viscometer, immersed in a temperature-controlled water bath. Viscometer calibration was based on water values and standard methods accepted by ASTM. A detailed description of this determination method is provided in prior publications (Harja *et al.*, 2020; Tataru – Fărnuș *et al.*, 2020).

3. Results and discussion

For the systems based on K_2CO_3 – KAla, with different concentrations of KAla (0, 3, 5, respectively 7%), the graph in Fig. 1 shows the variation of viscosity with temperature, at constant concentrations of the alanine salt. The potassium carbonate concentration was kept constant, 25% K_2CO_3 . From the same graphic representation, we can see the variation of viscosity with the concentration of potassium salt of alanine, the other parameters being constant (potassium carbonate and temperature).

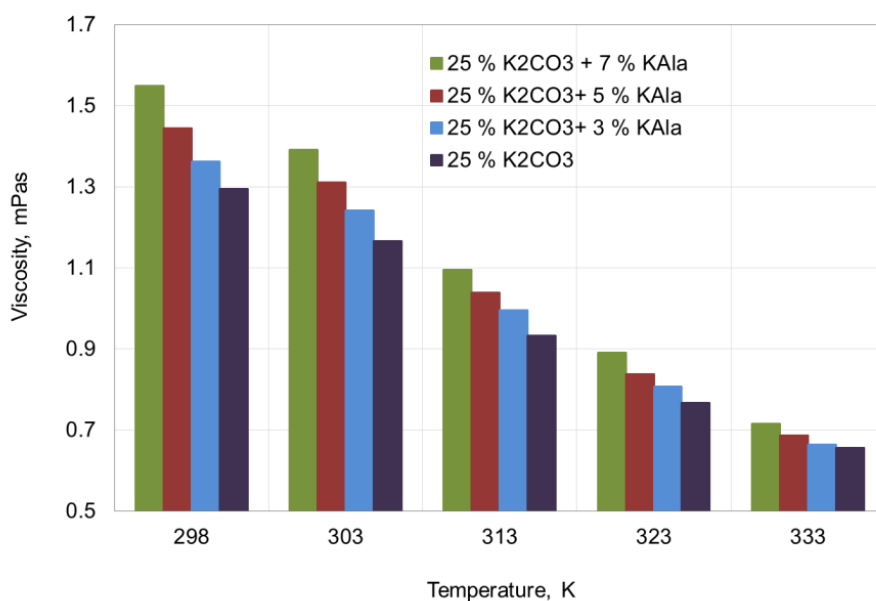


Fig. 1 – Variation of viscosity of solutions based on K_2CO_3 - KAla with temperature.

Viscosity is observed to continuously increase with concentration and decrease with increasing temperature for each system, as expected. This is because the internal resistance of the molecules decreases with increasing temperature, which allows the molecules of the solution to flow easily, thus

decreasing the viscosity. However, increasing the concentration of the potassium salt of alanine increases the viscosity of the mixture, due to the fact that molecular resistance increases in higher concentration solutions. At the temperature of 298 K and at the lowest mass fraction of 3% KAla, the viscosity value of the aqueous solution was 1.36 mPas. For the same temperature but maximum concentration in KAla (7%), the viscosity value is 1.55 mPas. When the temperature increases from 298 K to 333 K, the viscosity of K_2CO_3 -3% KAla solutions decrease by 49.32%, and in the case of K_2CO_3 -7% KAla solutions, the same increase in temperature causes a decrease in viscosity by 53.83%, which makes their application more practical at high temperature.

As can be seen from Fig. 1, there is a non-linear decrease in viscosity with increasing temperature. The experimental data include a wide variety of conditions (potassium carbonate concentration, temperature, amino acid salt concentration, etc.). A correlation of the viscosity values of these solutions is difficult because the properties of the liquid vary greatly, depending on the composition and temperature.

Statistic Approaches

The viscosity was predicted statistically as a function of temperature and the amount of KAla supplied. Regression analysis was the main emphasis of the employed technique, which was carried out using MINITAB 21. The goal was to choose the model that best fits the data and to create the ideal conditions for minimizing viscosity (process optimization) (Blaga *et al.*, 2022; Suditu *et al.*, 2022).

In this work, three feature selection techniques—stepwise (1), forward selection (2), and backward elimination (3)—were applied. The choice of features has a significant impact on how effectively the models work. The stepwise method begins with the null model and iterates through the following phases until no additional or removed independent variables are possible: To include a variable, a forward selection is employed first, followed by a reverse elimination. The most significant univariable model serves as the beginning model in the forward selection strategy. Then, until the list of variables is exhausted, the process of adding independent variables and model re-estimation is repeated. Starting with a global model, the backward elimination technique eliminates the most insignificant variables (and reevaluates the model) until no minor variables are left. In Table 1, the general statistics of the three methods used for the process under consideration are shown for an order 2 model with interactions through order 2 and complete cross predictors and 10-fold cross-validation.

Table 1
Statistics for the obtained models

	S	R-sq, %	R-sq (adj), %	PRESS	R-sq (pred), %	AICc	BIC	10-fold S	10-fold R-sq, %
(1)	0.0085	99.94	99.91	0.00254	99.83	-108.28	-117.32	0.011296	99.83
(2)	0.0081	99.95	99.91	0.00237	99.84	-103.35	-117.84	0.011226	99.83
(3)	0.0085	99.94	99.91	0.00254	99.83	-108.28	-117.32	0.011296	99.83

The Stepwise regression and Backward Elimination Regression produced the same statistical errors, as shown in Table 1. On closer examination, it was discovered that these two methods produced the identical model. Furthermore, there aren't many changes between forward selections and stepwise when comparing the statistics. However, as R-sq and R-sq(pred) are somewhat higher and S and PRESS are little lower, the forward selection is the optimal approach for the process under consideration. The analysis of variance for this forward selection regression is presented in Table 2, and Eq. (1) describes the model for uncoded variables.

Table 2
Analysis of variance

Source	DF	Seq SS	Contribution, %	Adj SS	Adj MS	F-Value	P-Value
Regression	8	1.45827	99.95	1.45827	0.182283	2773.27	0.000
(KAla)	1	0.03213	2.20	0.00147	0.001468	22.34	0.001
(Temp)	1	1.39560	95.66	0.00008	0.000079	1.20	0.297
(KAla) ²	1	0.00125	0.09	0.00003	0.000027	0.41	0.534
(Temp) ²	1	0.02111	1.45	0.00011	0.000110	1.67	0.223
(KAla)(Temp)	1	0.00643	0.44	0.00154	0.001541	23.44	0.001
(KAla) ³	1	0.00135	0.09	0.00135	0.001346	20.48	0.001
(Temp) ³	1	0.00014	0.01	0.00014	0.000139	2.12	0.174
(KAla) ² Temp	1	0.00026	0.02	0.00026	0.000260	3.96	0.072
Error	11	0.00072	0.05	0.00072	0.000066		
Total	19	1.45899	100.00				

$$\begin{aligned}
 \text{Viscosity} = & -45.1 + 0.2751 \cdot (\text{KAla}) + 0.557 \cdot (\text{Temp}) - 0.00544 (\text{KAla})^2 - \\
 & - 0.00209 \cdot (\text{Temp})^2 - 0.000891 \cdot (\text{KAla}) \cdot (\text{Temp}) - 0.001118 \cdot (\text{KAla})^3 \quad (1) \\
 & + 0.000002 (\text{Temp})^3 + 0.000051 \cdot (\text{KAla})^2 \cdot (\text{Temp})
 \end{aligned}$$

More detailed research of the variance revealed that the completed cross predictors contributed little to the model's overall performance. To lessen the

complexity of the model, the same forward selection technique was used on an order 2 model with only order 2 interactions. The statistic is shown in Table 3, and the analysis of variance is shown in Table 4.

Table 3
Statistics for the order 2 model with order 2 interactions

S	R-sq, %	R-sq (adj), %	PRESS	R-sq (pred), %	AICc	BIC	10-fold S	10-fold R-sq, %
0.01328	99.83	99.77	0.005078	99.65	-99.90	-102.27	0.017306	99.59

Table 4
Analysis of variance for order 2 model with order 2 interactions

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Regression	5	1.45652	99.83%	1.45652	0.291304	1651.81	0.000
(KAla)	1	0.03213	2.20%	0.00833	0.008327	47.22	0.000
(Temp)	1	1.39560	95.66%	0.02611	0.026110	148.05	0.000
(KAla) ²	1	0.00125	0.09%	0.00125	0.001251	7.09	0.019
(Temp) ²	1	0.02111	1.45%	0.02111	0.021109	119.70	0.000
(KAla)(Temp)	1	0.00643	0.44%	0.00643	0.006426	36.44	0.000
Error	14	0.00247	0.17%	0.00247	0.000176		
Total	19	1.45899	100.00%				

Tables 1 and 3 can be compared to see how the performance improved with the addition of full cross predictors. The model was more complex, though, and many of the terms had a P-value more than 0.05. As a result, the order 2 model with order 2 interactions and no cross predictors (Eq. 2) is taken into account in this work's optimization step. Five solutions were produced by the response optimizer to reduce viscosity (Table 5).

$$\text{Viscosity} = 32.80 + 0.1953 \cdot (\text{KAla}) + 0.1838 \cdot (\text{Temp}) - 0.001429 (\text{KAla})^2 - 0.000262 \cdot (\text{Temp})^2 - 0.000541 \cdot (\text{KAla}) \cdot (\text{Temp}) \quad (2)$$

Table 5
Response optimizer solutions

Solution	KAla	Temp	Viscosity Fit	Composite Desirability
1	0.00000	333.000	0.646097	1.00000
2	0.00000	331.968	0.656006	1.00000
3	3.50000	333.000	0.681202	0.96805
4	7.00000	333.000	0.681298	0.96793
5	3.89139	333.000	0.682951	0.96584

3. Conclusions

Despite the fact that amino acids (salts) are used in several industrial applications, little is known about these systems. Knowledge of the liquid viscosities are required for the solution of many engineering problems, including heat and mass transfer, and fluid flow. Viscosity values of amino acid salt – potassium carbonate solutions are needed for the design of gas-liquid contactors in the absorption process. In this work the physical properties of aqueous potassium carbonate solutions activated with the potassium salt of alanine were measured over a wide range of temperatures 298 K to 333 K. The correlations of the dimensionless parameters with temperature and KAla % concentration derived from the experimental data are in excellent agreement with the experimental data. In industrial applications, the generated models may offer a chance for real-time monitoring of absorbent composition, such as AAS concentration.

The proposed model is easy to apply and is of real help for the specialists in the field, to benefit from a reliable estimate of the viscosities of solvents based on potassium carbonate and alanine.

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STUDIUL PRIVIND VISCOZITATEA SOLVENȚILOR PE BAZĂ DE ALANINĂ ȘI CARBONAT DE POTASIU

(Rezumat)

Datorită energiei reduse necesare pentru regenerare, vitezei scăzute de degradare și corozivității mici, carbonatul de potasiu este un solvent excelent pentru captarea dioxidului de carbon. Cu toate acestea, carbonatul de potasiu prezintă un dezavantaj major prin faptul că are o viteză de reacție relativ lentă cu CO_2 , necesitând utilizarea unui utilaj de dimensiuni mari pentru procesul de absorbție. Adăugarea de promotori la soluțiile de carbonat de potasiu este cea mai eficientă modalitate de a crește cinetica de absorbție. Sarea de potasiu a alaninei (KAla) a fost aleasă ca promotor pentru absorbția CO_2 în soluție de carbonat de potasiu (25% K_2CO_3). Densitatea și vâscozitatea solvenților sunt cruciale pentru evaluarea lor amănunțită pentru aplicații industriale și ar trebui luate în considerare în plus față de date privind viteza de absorbție. Vâscozitatea acestor solvenți a fost evaluată în intervalul de temperatură de 298 - 333 K, pentru concentrații variind de la 3% KAla până la maxim 7% KAla. Vâscozitatea a fost prezisă statistic în funcție de temperatură și cantitatea de KAla furnizată. Analiza de regresie a fost principalul accent al tehnicii folosite, care a fost realizată cu MINITAB 21.