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SOLID-LIQUID EQUILIBRIUM AND PHASE DIAGRAM OF ORGANIC SYSTEM FORMING SIMPLE EUTECTIC

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ABSTRACT

The study of phase diagram guided with solid-liquid equilibrium (SLE) data at different molar composition in two component systems serve as a basic roadmap for the material synthesis, and also provide in-depth study of physicochemical and thermochemical properties of synthesized materials. The solid-liquid equilibrium data of binary organic system, acetanilide – acetamide (ACN-ACT) is determined by thaw melt method. The system exhibits simple eutectic type phase diagram at 0.20 molar composition of ACN with congruent melting temperature 52^{o}C.

Keywords: eutectic - phase diagram - solid-liquid equilibrium.

INTRODUCTION

The ever-growing demand of materials to cater the needs of modern civilization has been necessity for the search of novel materials having various specific properties and of low cost. Since the single component materials very often fail to meet the particular requirement of materials, property demanded, and therefore, the development of binary composite materials is of prime importance(Neupane, 2018). Phase diagram is a powerful tool for designing binary materials, by the combination of two different components, at different composition and thus formed binary shows excellent unique properties than its combining materials (Varukolu et al., 2021). The study of phase diagram guided with solid-liquid equilibrium (SLE) data at different molar composition in two component systems serve as a basic roadmap for the material synthesis, and also provide in-depth

study of physicochemical and thermochemical properties of synthesized materials(Sangster, 1999; Sharma & Rai, 2012). Further, a knowledge of SLE is of great technical importance to obtain a crystal of very high purity by designing and optimizing the crystallization process(Wittig & Constantinescu, 2001).

The organic compounds seem to be the potential candidate for the investigation of binary systems than that of metallic system due to their specific properties such as, low transformation temperature, transparency, wider choice of materials, low density driven convection effect, low cost, ease in purification and structural modification(Neupane & Rai, 2021).In the last few decades, the continuous efforts are being made in material science to discover the smart hybrid materials, often called as intermolecular compound, of interest by establishing phase diagram which give melting temperature and composition of the intermolecular compounds and eutectics. Most of the organic systems which are being recently studied result simple types eutectic mixture(Kim et al., 2006; Rai & Rai, 1998)rather than novel intermolecular compound(Rastogi, 1964). Eutectics find several applications in diverse fields of daily life. From the traditional refrigeration and snow removal (sodium chloride–water eutectic) and anti-freeze (ethylene glycol–water eutectic) in vehicles to the more recent energy storage devices, and from conventional soldering materials (lead–tin alloy) to novel materials in ceramics and glass industry, eutectics are present in day-to-day materials as well as in pharmaceutical formulations (Cherukuvada & Nangia, 2014; Karaipekli & Sari, 2010).

Herein, we report phase diagrams of two binary organic systems and their solid-liquid equilibrium data. The organic compounds; acetanilide (ACN) and acetamide (ACT), which are being used as active pharmaceutical ingredients are chosen for the study of phase diagram. ACN possesses analgesic as well as antipyretic properties, and is used as medical practice under the name of Antifebrin (Cahn, A., & Hepp, 1886). Similarly, ACT serves as a major ingredient in the synthesis of various pharmaceuticals, pesticides, and antioxidants(Ho et al., 2010).The binary phase diagram and solid-liquid equilibrium data of novel organic system; acetanilide-acetamide (ACN-ACT) is reported for the first time.

MATERIALS AND METHODOLOGY

The parent compounds, acetanilide (ACN) of 98% and acetamide (ACT) of 99%purity were purchased from Qualigen, India. The compounds were used as such

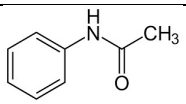
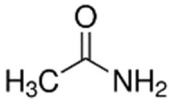
without further purification. The purity of each compound was accessed by melting point technique by comparing their experimental melting temperature with corresponding literature values (McGraw-, 1985).The melting point of parent compounds along with their purity information was summarized in Table 1.

The mixtures, covering the entire range of composition by weight, were taken in different proportion in well cleaned and dried long necked glass test tubes. The test tube containing the binary mixture was then covered with aluminum foil to avoid interaction with air and to prevent loss of material. Each test tube was individually put into paraffin oil bath maintained at a temperature about 10oC above the melting temperature of the higher melting component. The molten mixture was thoroughly shaken and suddenly quenched in ice cold water. The process of melting and quenching was repeated 4 times in order to ensure homogeneous mixing. The solidified mixture was then crushed and packed in vials and labelled. The melting temperature of each sample was determined by taking a pinch powdered mixture in a capillary tube sealed at ine end and kept in electrically operated Digital Melting Point Apparatus, which could read correctly up to ± 1.0^{o}C. Finally, a phase diagram was established by plotting mole fraction on X-axis and its corresponding melting temperature on Y-axis.

RESULTS AND DISCUSSION

The phase diagram of ACN-ACT systems is established by plotting the melting temperature versus composition expressed in mole fraction. The melting temperatures of all the parent compounds obtained in this study and those reported in literature are given in Table 1. The phase diagram ACN-ACT system reveals the formation of simple eutectic at 0.20 mole fraction of ACN (XACN) and its corresponding temperature is 52^{o}C (Figure 3). The solid-liquid equilibrium data in the entire range of composition for ACN-ACT system is reported in Table 2.

Table 1: The experimental thermodynamic parameters for the pure compounds (P = 101.3 KPa)[a] along with the literature data, and detail information of materials used in this work

Chemical name	Structure	Source	Molar mass (g mol^{-1})	Melting point T_m (K)		Purification method
				Experiment[b]	Literature[c]	
acetanilide (ACN)		Qualigen	135.16	112	114	none
acetamide (ACT)		Qualigen	60.06	79	79-81	none

Note: [a]The standard uncertainties u of p and T are u(p) = 5 kPa, u(T) = 0.5 K and equipment uncertainties (0.95 level of confidence). [b]The experimental melting temperatures are determined using digital melting point apparatus. [c]The literature values are taken from "Hand book of organic chemistry" (McGraw-, 1985)

Table 2: The solid-liquid equilibrium data of acetanilide-acetamide system forming simple eutectic.

S. N.	Mole fraction of acetanilide (X_{ACN})	Weight of acetanilide + acetamide $W_{ACN} + W_{ACT}$	Melting point (°C)
1.	0.00	0.000 + 1.000	79 (ACT)
2.	0.05	0.107+0.892	73
3.	0.10	0.202+0.797	65
4.	0.15	0.287+0.712	58
5.	0.20	0.363+0.636	52 (Eutectic)
6.	0.25	0.432+0.567	55
7.	0.30	0.495+0.505	58
8.	0.35	0.552+0.448	63
9.	0.40	0.604+0.396	65
10.	0.45	0.651+0.348	68
11.	0.50	0.695+0.304	72
12.	0.55	0.736+0.263	75
13.	0.60	0.774+0.225	78
14.	0.65	0.809+0.190	81
15.	0.70	0.842+0.157	84
16.	0.75	0.872+0.128	88
17.	0.80	0.901+0.099	93
18.	0.85	0.928+0.072	98
19.	0.90	0.953+0.047	102
20.	0.95	0.977+0.023	108
21.	1.00	1.000 +0.000	112 (ACN)

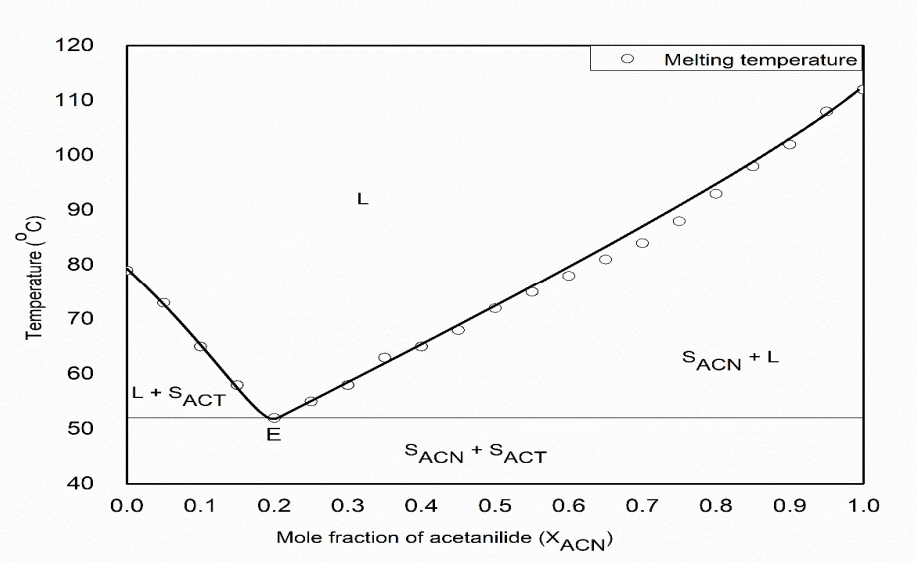


Figure 3: Phase diagram of acetanilide-acetamide system

CONCLUSION

The phase diagrams of binary systems, acetanilide – acetamide (ACN-ACT) is established by plotting the melting temperature versus mole fraction of components. The melting temperature of different components of mixture is determined by thaw melt method. The phase diagram of ACN-ACT system shows the formation of simple eutectic at 0.20 mole fraction of ACN. The eutectic mixture shows the congruent melting point of 52^0C. The current investigation has led importance knowledge about the eutectic composition between two organic compounds having active pharmaceutical ingredients.

REFERENCES

Cahn, A., & Hepp, P. . . (1886). Das antifebrin, ein neues fiebermittel. *Zentralblatt Fur Klinische Medizin*, *77*, 561-564.

Cherukuvada, S., & Nangia, A. (2014). Eutectics as improved pharmaceutical materials: Design, properties and characterization. *Chemical Communications*, *50*(8), 906–923.

https://doi.org/10.1039/c3cc47521b

Ho, T.-L., Fieser, M., Fieser, L., Danheiser, R., & Roush, W. (2010). Acetic anhydride. *Fieser and Fieser's Reagents for Organic Synthesis*, *1*, 146–160. https://doi.org/10.1002/9780471264194.fos00021.pub3

Karaipekli, A., & Sari, A. (2010). Preparation, thermal properties and thermal reliability of eutectic mixtures of fatty acids/expanded vermiculite as novel form-stable composites for energy storage. *Journal of Industrial and Engineering Chemistry*, *16*(5), 767–773. https://doi.org/10.1016/j.jiec.2010.07.003

Kim, H. J., Kim, J. H., Youn, S. H., & Shin, C. S. (2006). Thermodynamic and morphological analysis of eutectic formation of CBZ-l-Asp and l-PheOMe·HCl mixtures. *Thermochimica Acta*, *441*(2), 168–175. https://doi.org/10.1016/j.tca.2005.12.001

McGraw-, D. J. L. (1985). *Handbook of Chemistry*. New York: *McGraw- Hill*.

Neupane, U. (2018). Thermal , physicochemical and spectroscopic studies on some novel organic complexes obtained by green synthesis. *Journal of Thermal Analysis and Calorimetry*, *0123456789*, 1741–1752. https://doi.org/10.1007/s10973-018-7051-z

Neupane, U., & Rai, R. N. (2021). *Co-crystals formation specified from binary phase diagram studies; Their thermochemical, spectroscopic and optical properties*. https://doi.org/10.1016/j.jct.2021.106513

Rai, U. S., & Rai, R. N. (1998). Chemistry and Characterization of Binary Organic Eutectics. *Asian Journal of Chemistry*, *10*(3), 421–431.

Rastogi, R. P. (1964). Thermodynamics of phase equilibria and phase diagrams. *Journal of Chemical Education*, *41*(8), 443–448. https://doi.org/10.1021/ed041p443

Sangster, J. (1999). Phase diagrams and thermodynamic properties of binary systems of drugs. *Journal of Physical and Chemical Reference Data*, *28*(4), 889–930. https://doi.org/10.1063/1.556040

Sharma, K. P., & Rai, R. N. (2012). Thermochimica Acta Synthesis and characterization of novel binary organic monotectic and eutectic alloys. *Thermochimica Acta*, *535*, 66–70. https://doi.org/10.1016/j.tca.2012.02.020

Varukolu, M., Palnati, M., Nampally, V., Gangadhari, S., Vadluri, M., & Tigulla, P. (2021). New Charge Transfer Complex between 4-Dimethylaminopyridine and DDQ: Synthesis, Spectroscopic Characterization, DNA Binding Analysis, and Density Functional Theory (DFT)/Time-Dependent DFT/Natural Transition Orbital Studies. *ACS Omega*. https://doi.org/10.1021/acsomega.1c05464