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# Qualitative Inherent Safety Assessment for Flammability Evaluation of Deep Eutectic Solvent

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**Abstract**

Due to the increasing demand for green and sustainable analytical methods, many researchers have been using various attempts to improve and design environmentally friendly chemical processes. Based on the literature, most of the researchers agreed that deep eutectic solvent (DES) is one of the attractive solutions to environmentally friendly chemicals due to its classification as a green solvent and safe to use due to its low flammability characteristic. Although studies on the benefits and advantageous implementation of DESs are extensive, investigation on the safety level of DESs is still lacking. This paper aims to introduce a simple qualitative inherent safety assessment method for flammability evaluation of DESs. Flash point values of the HBA and HBD component of DES are used in evaluating the flammability level of the DES. Subjective score assignment was utilized based on the flash point data as well as mixing temperature in the production of DES. In this scoring method, higher score represents higher flammability hazard. The resulted scoring method has five level of hazards representing five range of flash point values with 5 indicates the highest and most hazardous score while 1 is the lowest and the least hazardous score. Implementation on a simple case study shows that this scoring method is easy to use and can be easily improved for future usage as more safety information on DES emerge.

Keywords: Deep Eutectic Solvents; Flammability; Inherent Safety Assessment; Flash Point; Hydrogen Bond Acceptor; Hydrogen Bond Donor.



## 1.0 INTRODUCTION

Increasing demand for green and sustainable analytical methods motivates numerous researchers in finding innovative ways to improve and design environmentally friendly chemical processes and one example of it is the development of deep eutectic solvent. Deep eutectic solvents (DESs) are homogeneous systems composed of two or more components [1]. Each DES is comprised of hydrogen bond acceptor (HBA) and hydrogen bond donor (HBD). There are various types of combinations between HBA and HBD in DES production with their own respective ratios. Most types of DESs could be prepared handily via simple mixing. Generally, DES can be obtained by heating the salt HBA and HBD at a certain molar ratio simultaneously at respective temperatures while stirring at a certain time producing colourless liquid DES.
It is an attractive alternative substitute to ionic liquids (ILs) due to their common characteristics with low cost of production. In addition, ILs are expensive for larger applications with low biodegradation as mentioned by Manurung et al. [2] which raise the attractiveness of DESs as its substitutes. According to Zhang et al. [3], DESs gain have been proven as biodegradable. DESs are a sustainable solvent that can help in reducing the dependency to hazardous organic solvents that are harmful to the human health as well as the environment as mentioned by Suhaili et al. [4]. The solvent has low volatility, low flammability, high biodegradability, non-toxic and is inexpensive. It has attracted considerable attention

in the area of synthesis, electrochemistry, materials, biochemistry, and separation. The DESs are used frequently in metal electropolishing [5], and metal electrodeposition [6].

The benefits of DES have been discussed in extensive amounts of publications. As an example, DESs have negligible volatility at room temperature, non-flammable, having high viscosity, environmentally benign and biodegradable [7]. This leads to DES being characterized as a green chemical based on the 12 Principles of Green Chemistry especially on the waste prevention, atom economy, less hazardous chemical synthesis, safer solvents and auxiliaries, and design for degradation according to Mamtani et al. [8]. Zhang et al. [3] mentioned in their work that DESs is not a volatile organic solvent and is not flammable, making their storage convenient in terms of the safety aspects. Both Vanda et al. [9] and Wu et al. [10] agree that DESs are categorized as low flammability solvent. This is also supported by Zhang et al. [3] and Gurkan et al. [11].

Although there is extensive research focusing on proving the health and environmental benefits of DES, discussion on the safety level of DES is quite lacking. Due to the lack of discussion on the safety level of DES as mentioned previously, this paper aims to introduce a simple qualitative inherent safety assessment method focusing on the flammability level of DES. The flammability level of DES is evaluated based on the flash point of HBA and HBD consist in each DES. A flash point is the lowest temperature at which a chemical can vaporize to form an ignitable mixture in air [12]. In short, the lower the value of flash point, the more flammable the chemical, in another word, it will be easier to burn. Throughout this study, there are some limitations in obtaining information regarding safety assessment on DESs as the extensive literatures are more focused on the preparation of DESs and its implementation. Therefore, the qualitative inherent safety assessment method introduced will utilize subjective scoring assignment based on the flash point data gathered and the temperature used in the production of DES.

## 2.0 METHODOLOGY

The qualitative inherent safety assessment method developed involves two main steps. The first step is data collection followed by the second step which is scoring development. The methodology of the research is represented by the flowchart as shown in Figure 1.

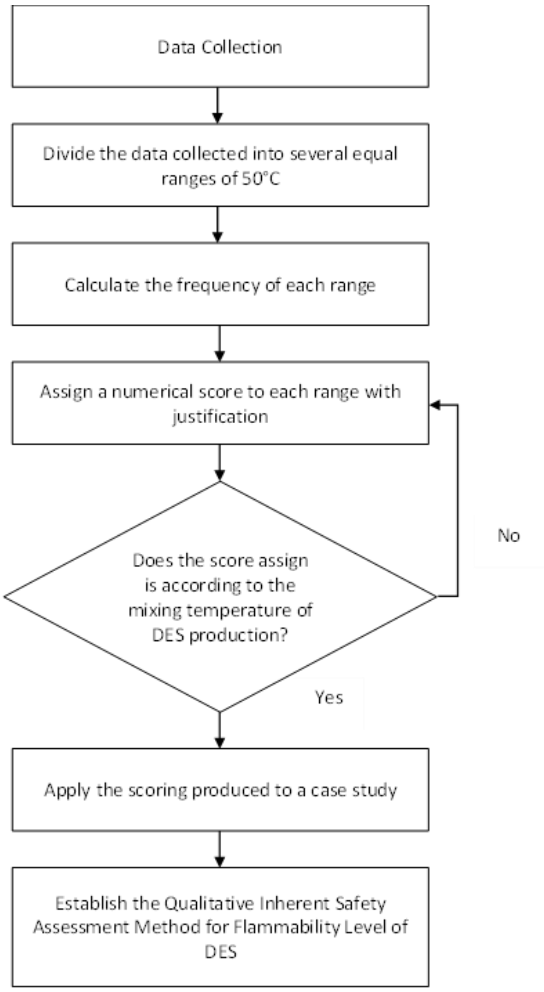


**Figure** 1: Methodology Flowchart

## 2.1 Data Collection

As mentioned previously, flash point is used in evaluating the flammability level of DES in this qualitative inherent safety assessment method. In this step, data collection focusing on the flash point values for available HBA and HBD is done. The identified HBA and HBD their respective boiling and flash points are as shown in Table 1.

**Table 1.** Flash Point Data for identified HBA and HBD

| Chemicals | Chemical Formula | Boiling Point (°C) | Flash Point (°C) |
|---|---|---|---|
| **Hydrogen Bond Acceptor (HBA)** | | | |
| Choline Chloride | ChCl | 100 | - |
| Zinc Chloride | $ZnCl_2$ | 732 | 732 |
| Tetrabutyl Ammonium Bromide | $C_{16}H_{36}BRN$ | 144.3 | 76 |
| Betaine | $C_5H_{11}NO_2$ | 218.95 | - |
| Menthol | $C_{10}H_{20}O$ | 212 | 93 |
| Tetraetyl-Ammonium Chloride | $C_8H_{20}CIN$ | 273.32 | - |
| Thymol | $C_{10}H_{14}O$ | 232 | 102 |
| Tetrabutyl-Ammonium Chloride | $(C_4H_9)_4NCI$ | - | 113 |
| Methyl Triphenyl-Phosphonium Chloride | $C_{19}H_{18}CIP$ | - | 250 |
| N-Methylmopholine-N-Oxide | $C_5H_{11}NO_2$ | 118 | 118 |
| Glycine | $C_2H_5NO_2$ | 233 | 176.67 |
| Methyl Triphenyl-Phosphonium Bromide | $C_{19}H_{18}BrP$ | - | 240 |
| Benzyltriphenylphosphonium Chloride | $C_{22}H_{22}CIP$ | - | 300 |
| Camphor | $C_{10}H_{16}O$ | 203.9 | 65 |
| **Hydrogen Bond Donor (HBD)** | | | |
| Benzamide | $C_7H_7NO$ | 180 | 180 |
| Ethylene Glycol | $C_2H_6O_2$ | 196 | 111 |
| Glycerol | $C_3H_8O_3$ | 290 | 193 |
| Acetamide | $C_2H_5NO$ | 221 | 1 |
| Benzoic Acid | $C_7H_6O_2$ | 249.2 | 121 |
| 1,3-Dimethyl Urea | $C_3H_8N_2O$ | 268 | 157 |
| Oxalic Acid | $C_2H_2O_4$ | 100 | - |
| Phenylacetic Acid | $C_8H_8O_2$ | 265 | 132 |
| Phenylpropionic Acid | $C_9H_{10}O_2$ | 260 | 148 |
| Malonic Acid | $C_3H_4O_4$ | 140 | 172 |
| Imidazole | $C_3N_2H_4$ | 255 | 145 |
| Adipic Acid | $C_6H_{10}O_4$ | 338 | 196 |
| Succinic Acid | $C_4H_6O_4$ | 235 | 110 |
| Tricarballytic Acid | $C_6H_8O_6$ | 227.7 | - |
| Levulinic Acid | $C_5H_8O_3$ | 245 | 137.8 |
| Itaconic Acid | $C_5H_6O_4$ | 268 | 268 |
| Xylitol | $C_5H_{12}O_5$ | 494.5 | 261.9 |
| D-Sorbitol | $C_6H_{14}O_6$ | 295 | 292.5 |
| 4-Hydroxybenzoic Acid | $C_7H_6O_3$ | 336 | 199 |
| Caffeic Acid | $C_9H_8O_4$ | 416.8 | 416.8 |
| Suberic Acid | $C_8H_{14}O_4$ | 230 | 203 |
| Gallic Acid | $C_7H_6O_5$ | 501.1 | 271 |
| Resorcinol | $C_6H_6O_2$ | 281 | 171.1 |
| Octanoic Acid | $C_8H_{16}O_2$ | 110 | 239.7 |
| Phenol | $C_6H_6O$ | 182 | 79 |
| 1,4-Butanediol | $C_4H_{10}O_2$ | 228 | 121 |
| Lactic Acid | $C_3H_6O_3$ | 122 | 112 |
| Diethylene Glycol | $C_3H_6O_3$ | 245 | 124 |
| Magnesium Chloride Hexahydrate | $MgCl_2.6H_2O$ | - | 1412 |
| 2,2,2-Trifluoroacetamide | $C_2H_2F_3NO$ | 162 | 163 |

## 2.2 Qualitative Scoring Development

The flash point data collected in the first step is then divided into several equal ranges of 50°C as shown in Table 2. The frequency of the data or also refers to the number of times the data value occurs in each range was calculated. Lastly, a score was assigned to each range with justifications on the flammable level for each range. According to the data analysis conducted as shown in Table 2, there is no HBA or HBD with flash point in the range of 0 until 50°C, 4 chemicals with flash point in the range of 51 - 100°C, 13 in the range of 101 - 150°C, 9 in the range of 151 - 200°C, and 12 chemicals with flash point value higher than 200°C.

Next, information on the mixing temperature of HBA and HBD to produce DES was also gathered to be used as justification in assigning the hazard level to the flash point range. In doing this, several assumptions were taken into consideration which are;

i. Chemicals with flash point in the range that is near to the ambient or room temperatures are assumed to be the most hazardous in terms of flammability parameter. This is because, the chemicals are prone to become flammable during the mixing process of the DES production. This is assumed to be worst-case scenario as it can give direct impact towards the handlers of the chemicals.
ii. According to the literature, the mixing process in the DES production is usually conducted between the room temperature up until 85°C. Therefore, chemicals with flash point nearing 85°C are also deemed as hazardous due to its likeliness to become flammable during the mixing process of the DES production. Table 2 listed the temperatures used in the mixing of HBA and HBD for DES production.
iii. Flash point range with most chemicals in it is deemed to be neutral in terms of hazard level as it is assumed that with a majority of chemicals with the flash point values, proper guidelines and standard operation procedures on how to use it without causing any fire should be in place and notably aware by the researchers.

**Table 2**: Data Analysis

| Chemical | Formula | Flash Point (°C) | Temperature Range (°C) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | 0 - 50 | 51 - 100 | 101 - 150 | 151 - 200 | 201 - 250 | 251 - 300 | >300 |
| Camphor | $C_{10}H_{16}O$ | 65 | | / | | | | | |
| Tetrabutyl Ammonium Bromide | $C_{16}H_{36}BRN$ | 76 | | / | | | | | |
| Phenol | $C_6H_6O$ | 79 | | / | | | | | |
| Menthol | $C_{10}H_{20}O$ | 93 | | / | | | | | |
| Thymol | $C_{10}H_{14}O$ | 102 | | | / | | | | |
| Succinic Acid | $C_4H_6O_4$ | 110 | | | / | | | | |
| Ethylene Glycol | $C_2H_6O_2$ | 111 | | | / | | | | |
| Lactic Acid | $C_3H_6O_3$ | 112 | | | / | | | | |
| Tetrabutyl-Ammonium Chloride | $(C_4H_9)_4NCI$ | 113 | | | / | | | | |
| N-Methylmopholine-N-Oxide | $C_5H_{11}NO_2$ | 118 | | | / | | | | |
| Benzoic Acid | $C_7H_6O_2$ | 121 | | | / | | | | |
| 1,4-Butanediol | $C_4H_{10}O_2$ | 121 | | | / | | | | |
| Diethylene Glycol | $C_3H_6O_3$ | 124 | | | / | | | | |
| Phenylacetic Acid | $C_8H_8O_2$ | 132 | | | / | | | | |
| Levulinic Acid | $C_5H_8O_3$ | 137.8 | | | / | | | | |
| Imidazole | $C_3N_2H_4$ | 145 | | | / | | | | |
| Phenylpropionic Acid | $C_9H_{10}O_2$ | 148 | | | / | | | | |
| 1,3-Dimethyl Urea | $C_3H_8N_2O$ | 157 | | | | / | | | |
| 2,2,2-Trifluoroacetamide | $C_2H_2F_3NO$ | 163 | | | | / | | | |
| Resorcinol | $C_6H_6O_2$ | 171.1 | | | | / | | | |
| Malonic Acid | $C_3H_4O_4$ | 172 | | | | / | | | |
| Glycine | $C_2H_5NO_2$ | 176.67 | | | | / | | | |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Benzamide | $C_7H_7NO$ | 180 | | | | / | | | |
| Glycerol | $C_3H_8O_3$ | 193 | | | | / | | | |
| Adipic Acid | $C_6H_{10}O_4$ | 196 | | | | / | | | |
| 4-Hydroxybenzoic Acid | $C_7H_6O_3$ | 199 | | | | / | | | |
| Suberic Acid | $C_8H_{14}O_4$ | 203 | | | | | / | | |
| Octanoic Acid | $C_8H_{16}O_2$ | 239.7 | | | | | / | | |
| Methyl Triphenyl-Phosphonium Bromide | $C_{19}H_{18}BrP$ | 240 | | | | | / | | |
| Methyl Triphenyl-Phosphonium Chloride | $C_{19}H_{18}CIP$ | 250 | | | | | / | | |
| Xylitol | $C_5H_{12}O_5$ | 261.9 | | | | | | / | |
| Itaconic Acid | $C_5H_6O_4$ | 268 | | | | | | / | |
| Gallic Acid | $C_7H_6O_5$ | 271 | | | | | | / | |
| D-Sorbitol | $C_6H_{14}O_6$ | 292.5 | | | | | | / | |
| Benzyltriphenylphosphonium Chloride | $C_{22}H_{22}CIP$ | 300 | | | | | | / | |
| Caffeic Acid | $C_9H_8O_4$ | 416.8 | | | | | | | / |
| Zinc Chloride | $ZnCl_2$ | 732 | | | | | | | / |
| Magnesium Chloride Hexahydrate | $MgCl_2.6H_2O$ | 1412 | | | | | | | / |
| **Frequency** | | | **0** | **4** | **13** | **9** | **4** | **5** | **3** |

**Table 2**: Temperature Used in the Mixing of HBA and HBD in the Production of DES

| Temperature | Sources |
|---|---|
| 27 | Zhang et al [3]<br>Manurung et al [2] |
| 50 | Socas-Rodriguez et al [13] |
| 80 | Suhaili et al [4] |
| 85 | Socas-Rodriguez et al [13] |

## 3.0 RESULTS AND DISCUSSION

In this work, a qualitative inherent safety assessment method for flammability evaluation of DES was developed. Table 3 shows the scoring table produced for flash point evaluation. According to Table 3, there are five hazardous levels each assigned with a numerical score representing the level. In this scoring method, higher score represents higher hazard.

The first flash point range is between 0 to 50°C which is indicated as the extremely hazardous range represented with the highest score of 5. This range is deemed as extremely hazardous as it is the range that is the closest to the ambient and room temperature and it is deemed that the chemicals is prone to become flammable during the mixing process of the DES production with direct impact towards the handlers of the chemicals.

The second flash point range is between 51 – 100°C. This range is assigned as the hazardous with lesser score than the first range which is 4. The justification made to this hazardous level assignment is due to some process of DES production with HBA and HBD mixing temperatures of 80 to 85°C. According to the literature, the mixing process in the DES production is usually conducted between the room temperature up until 85°C. Therefore, chemicals with flash point nearing 85°C are also deemed as hazardous due to its proneness to become flammable during the mixing process of the DES production.

The next range is assigned as the neutral range with a temperature range of 101 - 150°C with a score of 3. This range is deemed as neutral due to the fact that the range is outside the common mixing temperature in the DES production which is 85°C. In addition, based on the data gathered in this work, most of the chemicals identified have a flash point value in this range. Flash point range with a majority of chemicals in it is deemed to be neutral in terms of hazard level as it is assumed that with a majority of chemicals with the flash point values, proper guidelines and standard operation procedures on how to use it without causing any fire should be in place and notably aware by the researchers.

The fourth level is the not hazardous level with a score of 2. The range of flash point temperature for this level is 151 - 200°C. This temperature range is far outside the common mixing temperature in the DES production and therefore is assigned as a not hazardous level compared to the third level. This assumption is also used in assigning the score for the last level which is the safe level with flash point temperature of higher than 200°C represented by a score of 1.

**Table 3:** Qualitative Inherent Safety Assessment Scoring for Flammability Evaluation of DES

| **Flash Point Range (°C)** | **0 - 50** | **51 - 100** | **101 - 150** | **151 - 200** | **201 - 250** |
|---|---|---|---|---|---|
| **Hazardous Level Assigned** | Extremely Hazardous | Hazardous | Neutral | Not Hazardous | Safe |
| **Score** | 5 | 4 | 3 | 2 | 1 |

## 4.0 APPLICATION OF THE METHOD DEVELOPED TO CASE STUDY

In order to illustrate the usage of the developed qualitative inherent safety assessment method for flammability evaluation of DES, a simple case study was used to evaluate the flammability level of three DES. Three DESs assigned as DES 1, DES 2, and DES 3 were evaluated for its flammability level as shown in Table 4. DES 1 is made of tetrabutyl ammonium bromide as HBA and ethylene glycol as HBD, DES 2 is made of menthol as HBA and 1,4-butandiol as HBD, while DES 3 is made of camphor as HBA and phenol as HBD. These DESs are usually used as a substitute to ionic liquids due to their biodegradability, cheap prices, and easy to prepare. According to Table 4, DES 3 is the most hazardous DES in terms of flammability due to the low flash point temperature of its HBA and HBD. This is due to the high score of flash point for camphor and phenol which is 4. Meanwhile, DES 1 and DES 2 have similar level of flammability hazard but are indicated as having lower hazard than DES 3. This can be seen in the lower score of 3 of one of their components which is ethylene glycol and 1,4-butanediol for DES 1 and DES 2, respectively. As the scoring method take the total score of both HBA and HBD in the DES as the representative, DES 1 and DES 2 are deemed as having less hazard in terms of flammability compared to DES 3.

The utilization of this scoring method is simple and easy-to-use. Aside from that, the scoring method can be improved on a timely basis as the data on the flash points values and amount of HBA and HBD improves indicating its flexibility for future enhancements. In addition, as the scoring method was developed subjectively according to several assumptions, the scoring can be improved in the future as more data on safety assessment of DES emerge.

**Table 4.** Utilization of the Developed Qualitative Inherent Safety Assessment Method on a Simple Case Study

| DES | Chemicals | Role | Flash Point (°C) | Score | Total Score | Rank |
|---|---|---|---|---|---|---|
| 1 | Tetrabutyl Ammonium Bromide | HBA | 76 | 4 | 7 | 2 |
| | Ethylene Glycol | HBD | 111 | 3 | | |
| 2 | Menthol | HBA | 93 | 4 | 7 | 2 |
| | 1,4-butanediol | HBD | 121 | 3 | | |
| 3 | Camphor | HBA | 65 | 4 | 8 | 1 |
| | Phenol | HBD | 79 | 4 | | |

## 5.0 CONCLUSION

In conclusion, this paper aims to introduce a qualitative inherent safety assessment method for flammability evaluation of DES. Flash point values of the HBA and HBD component of DES are used in evaluating the flammability level of the DES. Subjective score assignment was utilized based on the flash point data as well as mixing temperature in the production of DES. In this scoring method, higher score represents higher flammability hazard. There are five level of hazard representing five range of flash point values. This scoring method is easy to use and can be easily improved for future usage as more safety information on DES emerge. However, a more comprehensive inherent safety assessment of DES is needed focusing on explosiveness as well as toxicity parameters alongside flammability parameter. In addition, more factors need to be included in constructing the scoring method alongside mixing temperature for example storage condition. Inclusion of various factors and parameters will not only results in comprehensive inherent safety assessment, but also inherent safety assessment with higher accuracy.

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