

A Study on the Flash Point and Spontaneous Ignition Determination of Diethylene Glycol

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Abstract : This study investigates the main factors of fire prevention, such as spontaneous ignition and flash point, while using Diethylene Glycol (DEG) as the antifreeze for automobile and industrial machines. Our results show the flash point of 142°C and the minimum ignition temperature of 388°C in the range of 130 μ l ~ 150 μ l. By increasing temperature to ignite in 1 second, an instantaneous ignition temperature of the sample is 569°C in the sample amount of 140 μ l.

Key Words : Spontaneous ignition, Minimum ignition temperatures, Instantaneous ignition temperature

1. Introduction

The industrial society of today develops, synthesizes, and produces new chemical substances at an incredibly fast rate; in addition, the chemical industry becomes more massive and diversified. Due to the rapid economic growth, energy usage in all areas has experienced a dramatic increase. Due to the increase of manufacturing, storing, transferring, and using chemical materials like flammable gases and liquids, a fire or

explosion may accidentally occur anytime. Therefore, the prevention of a fire and explosion is an issue of paramount importance [1][2].

The flash point has been used as the index that shows risk of a fire and explosion. The flash point defines the minimum temperature of diverging steam in necessary concentration that is ignited near a surface of flammable liquid. The flash point consists of the lower and the upper flash points. Usually, the lower flash point

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is treated as the flash point and the upper flash point defines the temperature of the liquid where the saturated concentration of steam is equivalent to the upper explosive limit. An additional characteristic here is the so-called spontaneous ignition that helps to evaluate the risk of a fire and explosion. The spontaneous ignition is a natural combustion phenomenon caused by a substance's gradual reaching the ignition point when accumulating heat in the condition of room temperature and no external ignition source [3].

In the case of pure substance, physicochemical characteristics, like the flash point and spontaneous ignition point, are provided in the Material Safety Data Sheet (MSDS); however, the values may vary across different documents for the same substance [4]. For that reason, numerous difficulties arise with respect to treating dangerous substances in a workplace where chemical substances are used. Therefore, in order to prevent disasters in workplace, it is crucial to figure out the exact combustion characteristics of using substances, such as the flash point and spontaneous ignition temperature.

This study measured spontaneous ignition characteristics and the flash point of Diehylene Glycol (hereafter DEG) that is widely used for ink printing ink, as well as s break material, plasticizer, softener of cellophane, and antifreeze. Our ultimate aim was to provide basic guidelines for fire and explosion prevention in workplaces where

DEG is used [5].

2. Theoretical background

Spontaneous ignition is caused by a speedy unbalance of internal heat radiation and exothermic reaction during the heat-storing process. To achieve exothermic reaction, the calefaction temperature should be sufficiently high in the condition of high heat radiation. In the opposite case, when the heat radiation is low, then, an exothermic reaction occurs in low temperature.

In other words, the ignition is a transient phenomenon where a substance discontinuously transforms its combustion status from non-combusted status. Therefore, the ignition is always an abnormal problem and certain conditions of substance and energy must be satisfied to have the above transformation. Normally, it is called the limit of ignition. The combustion limit (Explosion limit) is used as an index of the condition of the substance and the ignition temperature and ignition energy are used convenient indexes of the condition of energy.

Generally, applying the Semenov thermal ignition theory to spontaneous ignition of liquid should be appropriate. The thermal ignition theory is a theory that explains the problems of balance between heat emission by conduction, convection, and radiation with the heat generated by an exothermic reaction. In addition, it is the Semenov theory that applies calculation of limitations during ignition caused by the speedy

unbalance of exothermic reaction and heat radiation. The spontaneous ignition is caused by the speed of exothermic reaction from the chemical reaction and the speed of heat radiation towards external system (see Fig. 1).

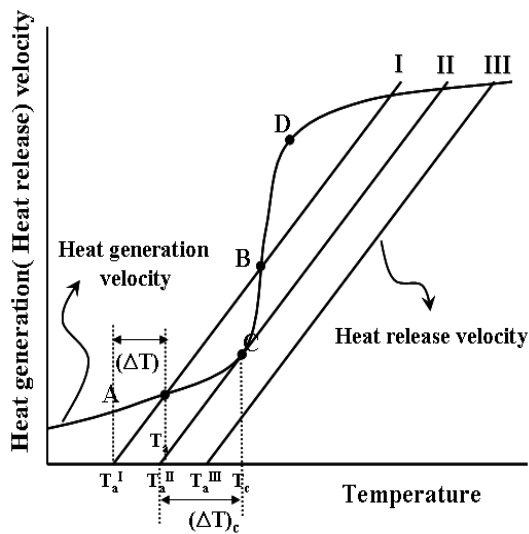


Fig. 1 Relation between heat generation and heat release in autoignition.

In other words, when the heating temperature is as low as T_a^I , the straight line is crossed with the curved line at point A, so that both gradually converge into a certain value. In this respect, if the heating temperature is as high as T_a^{III} , exothermic speed is above the heat radiation speed, unrelatedly to the temperature, so there must be ignition. Therefore, the limit of ignition is when the curved line contacts the straight line that is the case of T_a^{II} and point C is the maximum value just before from an ignition. During the induction period of ignition, fuel consumption is not

significant, so it was ignored. Also, it was assumed that there was no reaction in the high exothermic amount inside. Therefore, the temperature of the substance sustained as T until the reactant was completely combusted. In the assumption of the substance's uniform temperature when reactant liquid was sufficiently stirred in phenomenologically and heat radiation towards wall of container is occurred by convection [6].

3. Experiment

3.1 Sample

In this study, 99% pure and genuine first-grade DEG from JUNSEI was used as the sample; the physical and chemical properties of DEG are shown in Table 1 [7].

Table 1 Physical and chemical properties of DEG

Boiling point	244 ~ 245°C
Flammability	1.8% ~ 12.2%
Vapor pressure (at 25°C)	0.0057mmHg
Vapor density	3.66
Flash point	124°C
Auto ignition temperature	229°C
Viscosity (at 25°C)	30cP
Specific gravity (at 15°C)	1.1197
Molecular weight	106.12

3.2 Equipment

3.2.1 Flash point test equipment

The method of measuring the flash point is regulated in ASTM D1310-14 and, generally, it has the standard of 80°C. For above the standard, an open system is used.

For the temperature lower than standard, a closed system is used to keep the high accuracy of material properties.

Fig. 2 shows the photograph of the Tag open-cup apparatus above the experiment.

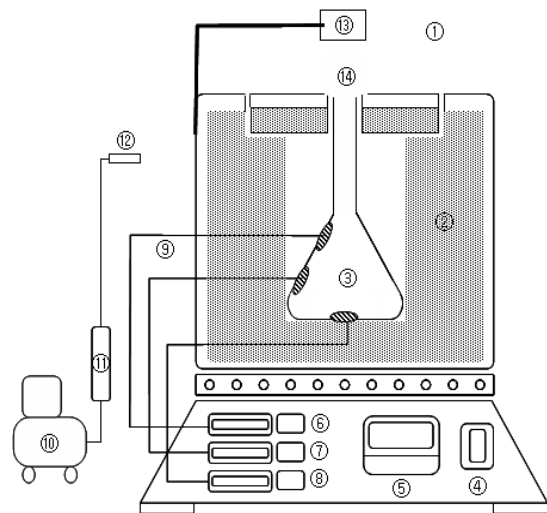


Fig. 2 Photograph of Tag open cup apparatus.

This study used the Tag open system for the experiment. The test cup was divided into the outer cup and the inner cup. The inner cup was a cylinder shape. There was a double heater between the outer and the inner cup to heat the sample container. The main body was made of a heating device, a power switch, electric heating controller, etc. The test flame applicator consisted of fuel tank, flame controller, gas pipe, and gas safety valve. It was possible to observe an ignition of the sample by contacting the test flame [8].

3.2.2 Spontaneous ignition point test equipment

This study used ASTM D2155-63T type equipment that has been popular for measuring the spontaneous ignition point. This method is standardized to check liquid petroleum products [9]. The sample was dropped into a container heated to a certain temperature and the ignition temperature was measured.



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|------------------------------|------------------------------|
| ① Electric furnace | ⑧ Lower part temp. indicator |
| ② Heater | ⑨ Air compressor |
| ③ Flask | ⑩ Thermocouple |
| ④ Power supply | ⑪ Silicagel column |
| ⑤ temperature controller | ⑫ Cleaning air tube |
| ⑥ Upper part temp. indicator | ⑬ Mirror |
| ⑦ Side part temp. indicator | ⑭ Sample injection port |

Fig. 3 Schematic diagram of experimental apparatus for spontaneous ignition measurement.

Fig. 3 shows the schematic diagram of the experiment.

To check spontaneous ignition of the liquid, 250ml of Pyrex triangular flask was fixed in the electric furnace and it was used as the measuring container. 0.3mm of Chromel-Alumei thermocouple was stuck

into the top, side, and bottom of the flask. Also, a reflector was installed on the top of the container to observe an ignition and an air compressor had been used to substitute steam inside of the measuring container. 250 μ l and 500 μ l micro cylinder from Hamilton, USA, was used for the collection of the sample amount and ignition delay time was measured with a micro-chronometer from Kappa that can measure 1/100 second [10].

4. Results and Discussion

4.1 Flash point

The flash point was measured by contacting the test flame with the sample and increase 1 $^{\circ}$ C after 1 minute. The method used the tag open system test equipment by following the method from ASTM D1310-14. The average flash point from the results from 5 trials of 100% DEG flash point (143 $^{\circ}$ C, 142 $^{\circ}$ C, 142 $^{\circ}$ C, 141 $^{\circ}$ C, 142 $^{\circ}$ C) was 142 $^{\circ}$ C.

4.2 Spontaneous ignition point

To get the minimum ignition temperature of 100% DEG, the temperature of 5 μ l ~ 350 μ l sample amount was changed to get the ignition limit temperature for each sample (see Fig.4).

The minimum ignition temperature of 100% DEG was 388 $^{\circ}$ C in the range of 130 μ l ~ 150 μ l. For over 449 $^{\circ}$ C, continuous ignition was observed at least twice when the ample amount was 145 μ l. That happened in 469 $^{\circ}$ C at 60 μ l. By increasing the temperature,

the igniting amount of the sample increased, when the temperature reached to 499 $^{\circ}$ C and, even in a low sample amount, ignition was observed at least two times. It seemed that the continuous ignition occurrence was caused from the scattering of separation of the droplet by surface boiling.

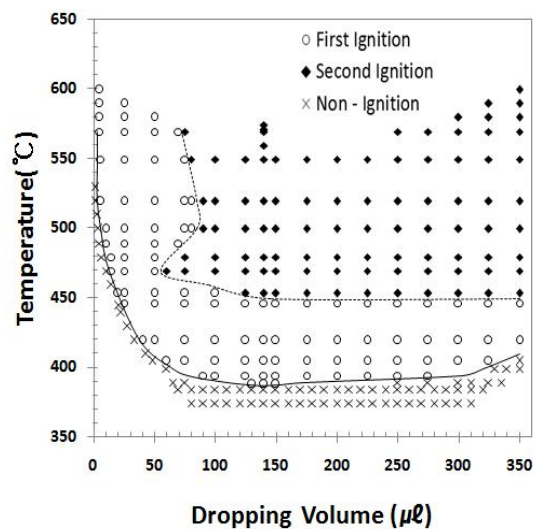


Fig. 4 Relative diagram between ignition temperature and dropping volume for DEG 100%.

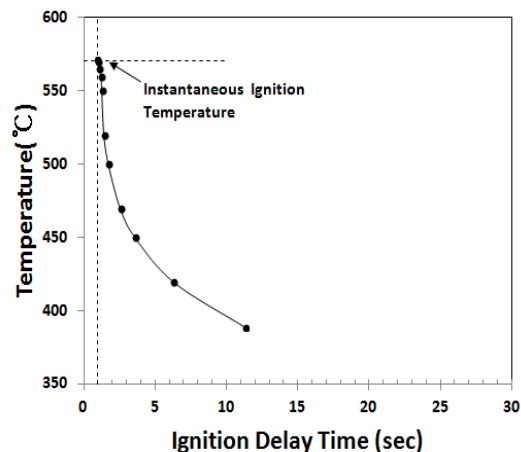


Fig. 5 Relative diagram between ignition delay time and ignition temperature for DEG 100% at 140 μ l.

4.3 Instantaneous ignition temperature

It was possible to get 388°C as the minimum ignition temperature of 100% DEG in 140 μ l. From the minimum ignition temperature at the sample amount of 140 μ l, the sample started to gradually ignite faster by increasing the temperature of the container after inserting the sample. It was possible to get 569°C as instantaneous ignition temperature when the ignition delay time was 1.0 second.

5. Conclusion

On considering the ignition characteristics of the DEG sample by using ASTM D2155-63T ignition temperature measuring equipment and the flash point measuring equipment, the following conclusions can be made.

- (1) The average flash point of the DEG sample amounted to 142°C.
- (2) The spontaneous ignition temperature of the DEG sample was 388°C in the range of 130 μ l ~ 150 μ l.
- (3) When the sample amount of DEG was more than 145 μ l and the temperature was 449°C, it was possible to observe continuous ignition at least 2 times.
- (4) Instantaneous ignition temperature of the DEG sample amounted to 569°C.

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