



Characterization of Polyglycol-Based Frothers: Investigation of Dynamic Froth Stability and Dynamic Frothability

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Abstract

Assessing frother performance through various indices is crucial to understanding how their molecular structure affects functionality, as well as evaluating their effectiveness in floating both fine and coarse particles. This study investigates for the first time the frothing behavior and froth stability of Polyethylene Glycol 300 (PEG300), Dipropylene Glycol (DPG), and Tetraethylene Glycol (TEG) and compares them with conventional frothers such as Dow Froth-250 (DF-250). To evaluate frother performance, air flow rate and frother concentration were selected as the main operational variables influencing froth formation and stability index. Initially, the frothing behavior of the reagents was predicted using the HLB-MW diagram, and then the frothing power of the desired frothers was examined using the dynamic frothability and dynamic froth stability indices. The results revealed that PEG300 exhibited the highest dynamic frothing index (13000 s.dm³/mol) and high froth stability, which is suitable for the flotation of coarse particles. In contrast, DPG showed the lowest frothing power and froth stability, with a dynamic frothing index of 2500 s.dm³/mol. TEG, with an intermediate frothing index of 5000 s.dm³/mol, demonstrated moderate performance in both froth production and stability. DF-250, with an exceptionally high frothing index, outperformed all the other agents, providing both superior froth generation and stability. Froth stability was assessed using dynamic froth stability indices and dynamic frothing capability, providing meaningful insights into frother performance. The results also showed that both air flow rate and frother concentration had a significant impact on frothing index and stability, with higher concentrations generally enhancing froth stability, particularly for PEG300 and DF-250.

1. Introduction

Flotation, which is widely used for the Beneficiation of fine particles, is based on the adsorption of aqueous particles by air bubbles. In this process, surface-activating agents, such as frothers, are used to help produce fine air bubbles and stabilize the froth, which facilitates the transport of particles by adsorption at the air-water interface. Frothers are surface-active compounds that consist of a polar group (OH, COOH, C=O, OSO₂, and SO₂OH) and a hydrocarbon chain [1, 2]. These compounds are active at the interface of liquid and gas; their connection mechanism is physical. The surface activity of a surfactant with a normal alkyl chain in its molecule increases by 3.2 times due to the addition of each -CH₂- group in the

molecule [3, 4]. The frother molecules are oriented at the air-water interface in such a way that the polar or hydrophilic group is directed towards the liquid and the non-polar hydrocarbon chain is directed towards the air. Frothers not only create a relatively stable froth but also produce small bubbles, and in this way, the increase and dispersion of air bubbles on the surface of the pulp is the responsibility of the frother. Frothers also control the shape of the air bubble, and the air bubble in the presence of the frother is more spherical and has a slower rising speed [2, 5-8]. The frothers have different structural groups, and the performance of frothers depends on their chemical structure. The use of frothers in mineral flotation



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practice today is dominated by two nonionic surfactant families, alcohols (general formula: $C_nH_{2n+1}OH$) and polyglycols such as PEO (polyethylene oxide), PPO (polypropylene oxide), and PBO (polybutylene oxide). The compounds can be expressed using the general equation $R(X)_nOH$, where R is H or C_nH_{2n+1} , and X is ethylene oxide (EO), propylene oxide (PO), or butylene oxide (BO). The presence of ether oxygen and hydroxyl groups imparts hydrophilic characteristics, whereas the propylene and butylene segments exhibit hydrophobic properties. The equilibrium between the hydrophobic and hydrophilic components in these substances is regulated by modifying the number of units within the alkyl ether and by altering the number of ethylene oxide (EO), propylene oxide (PO), or butylene oxide (BO) groups within the polyethylene oxide (PEO) chain[1, 2, 9-15]. Glycol-based frothers produce a relatively thick, stable froth with low selectivity, carry more water (i.e., are wet), and are less sensitive to pH changes[11, 16]. This property of polyglycols is considered an advantage when recovering coarse particles. The molecular weight and hydrocarbon chain length of the polyglycol ethers determine their frothing ability. Frothers with higher molecular weight produce a more stable froth but with lower selectivity[3, 9, 17-19]. These surfactants rank among the most adaptable neutral frothers and are likely the second most prevalent category of commercial frothers currently in use. Based on these functions, frothers can be categorized into two distinct types: selective and powerful. The term "selective" pertains to the flotation of fine particles, while the term "powerful" refers to the frothing capability, which is crucial for the recovery and efficiency of flotation processes involving coarse particles. Consequently, by employing suitable indices, one can evaluate the performance of frothers according to their varying structural characteristics. Frothers belonging to the alcohol groups behave more selectively and are, therefore, more suitable for the flotation of fine particles, while polyglycerols have

greater frothing power and are, therefore, suitable for the flotation of coarse particles[1, 11, 20-24].

One of the key parameters that reflects the amphiphilic nature of frothers is the Hydrophilic-Lipophilic Balance (HLB), first proposed by Griffin and later refined by Davies. HLB values, typically ranging from 0 (fully hydrophobic) to 20 (fully hydrophilic), provide insight into the balance of polar and non-polar groups in a molecule. The Davies method estimates HLB by assigning group numbers to hydrophilic and lipophilic functional groups within a compound. This structural parameter is frequently used to predict surfactant behavior in aqueous systems, including flotation[25-32]. The critical role of froth stability in flotation is increasingly recognized, as it directly affects mineral grade determination and flotation recovery. The term froth stability is defined as the ability of froth bubbles to resist coalescence and collapse. In other words, a more stable froth has fewer coalescence and collapse events[33-35]. In this study, the Dynamic Frothability Index (DFI) and Dynamic Froth Stability Index (DFS) are used to investigate the stability of the froths of interest. DFI was described as a definitive measure of the stability of frother under dynamic conditions. DFI is used alongside static frothability as an acceptable criterion for measuring the properties of frothers. DFI is a characteristic of each frother. DFS, which is characterized by the ratio of froth volume to airflow in the system, serves as a metric for assessing froth stability in mineral flotation processes. [36-44] In this study, the effect of the molecular structure of frothers on their performance is determined through the indices of HLB, DFI, and DFS using frothing indices; the selectivity or power of the frothers can be investigated.

2. Material and Methods

2.1. Material

The frothers tested are listed in Table 1, selected to cover a range of molecular weights. All were reagent grade from Sigma-Aldrich (identified as 99% purity or higher).

Table 1. Frothers used in the study [20, 45, 46]

Frother type	Chemical formula	Molecular weight (g/mol)	HLB
Tetraethylene glycol	$H(C_2H_4O)_4OH$	194	12.1
PEG 300	$H(C_2H_4O)_6.4OH$	300	12.9
Dipropylene glycol	$H(C_3H_6O)_2OH$	134	9.25
DF-250	$CH_3(C_3H_6O)_4OH$	264.37	7.8

2.2. Methods

2.2.1. Hydrophilic-lipophilic balance

After Griffin, many attempts were made to provide a simple and repeatable method for calculating HLB, among which the Davis method is still the most widely used. [25, 26, 29-32]. The

HLB value for a particular frother can be ascertained by analyzing the types and quantities of functional groups present within the molecule, with each functional group corresponding to a designated group number[28]. In Davies' approach, the HLB is expressed by Equation 1.

$$\text{HLB} = 7 + \Sigma (\text{hydrophilic group numbers}) + \Sigma (\text{lipophilic group numbers}) \quad (1)$$

The HLB value calculated by the Davis method for PEG 300, DPG, and TEG frothers is 12.9, 9.25, and 12.1, respectively. The surfactants with lower HLB values are more hydrophobic than those with higher HLB values. In other words, surfactants with higher HLB are of more water solubility.

2.2.2. Prediction of frothing behavior

The molecular weight (M_w) of frothers plays a crucial role in flotation kinetics. Higher molecular weight frothers are known to produce more stable froth compared to their lower molecular weight counterparts. polyglycol-based frothers, which have larger molecular weights, are capable of floating larger particles and are more effective in floating a broader range of particle sizes, improving flotation recovery. Since higher molecular weight frothers reduce the rising bubble

velocity more, it is predicted that the frothers studied in this study will have higher frothing power than MIBC [1, 22, 47-54]. A popular method for describing a frother's power and selectivity is the HLB-Mw diagram. As can be observed, the frothers on the left side of this diagram are known to be selective in flotation, while the ones off to the right of this line are known to have strong flotation characteristics.[8, 20, 55-57]. From the data given in Table 1, the position of PEG300, TEG, and DPG frothers in the HLB-Mw diagram can be drawn (Figure 1). As seen in Fig, representative dotted for studied frothers is between Selective- and powerful lines, which means that may show an intermediate behavior in aqueous solution. It shows that all three frothers are more powerful than alcohol-based frothers and should give more frothing power. In contrast, they have less frothing power than DF-250 and would give lower frothability.

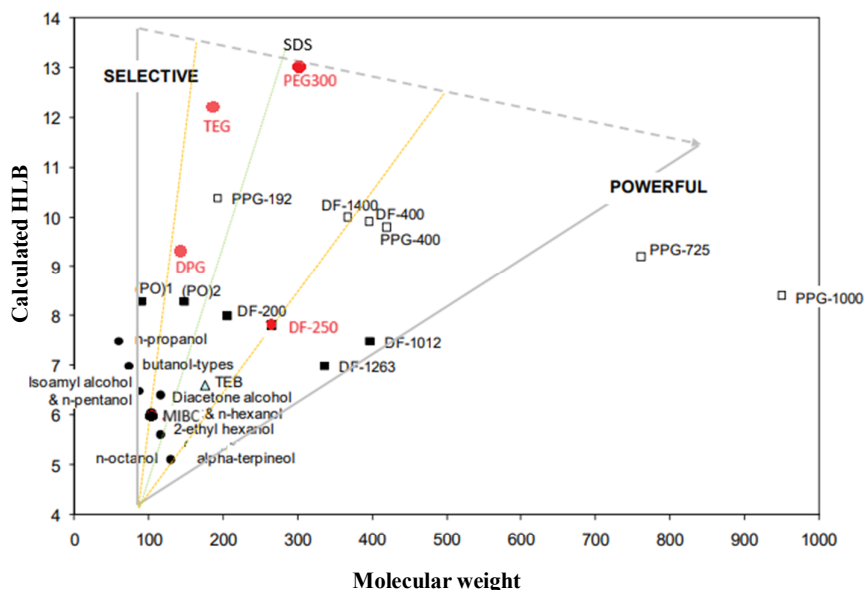


Figure 1. The prediction of frothability using HLB-Mw diagram[20]

2.2.3. Dynamic frothability index

The frothability tests were carried out using a froth column meter of 50 mm interior diameter and a glass cylindrical tube of 1000 mm height. The froth was generated by aerating the surfactant solution using a fritted glass sparger through a semi-permeable mesh screen with a pore size of 85 mesh (160 microns) at the bottom of the froth measurement column (Figure2). To start with the test, the froth column was filled with 200 mL of surfactant solution with concentrations of 5, 10, 15, 25, 50 and 100 ppm of frother. The flow meter was set to a determined air flow rate range of 1– 4 L/min, and when the froth height reached the equilibrium, it was recorded. Deionized water was used to create aqueous solutions of the examined frothers, and all tests were conducted at room temperature (25 ± 1 °C) and constant pH (pH: 7).

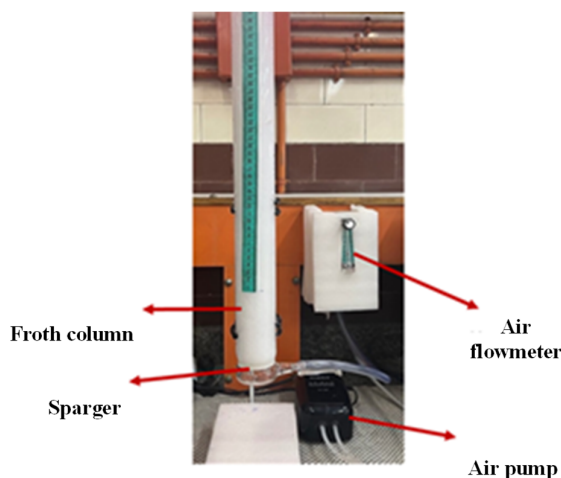


Figure 2. Setup of a froth column

The calculation of the DFI for a given frother involves plotting froth volume against the aeration rate, where the slope of this graph indicates the retention time of the froth (equation 2).

$$rt = \frac{\Delta V}{\Delta Q} \quad (2)$$

Where are:

rt - the froth retention time (s),

V - the gas volume (cm^3),

Q - the Air flow rate (cm^3/s).

To calculate the DFI, the values of rt are plotted against the corresponding concentration, and the slope of the linear portion of the resulting graph is equal to the DFI which is calculated using the equation (3)

$$DFI = \left(\frac{\partial rt}{\partial c} \right)_{c \rightarrow 0} \quad (3)$$

Frother that has a higher DFI is capable of producing a more voluminous froth with greater stability [20, 45, 58, 59].

2.2.4. Dynamic froth stability index

Dynamic froth stability is closely related to DFI. The evaluation of dynamic froth stability involves measuring the froth growth rate and the maximum equilibrium height under different airflow velocities and varying concentrations of froth stabilizers. The dynamic stability index is affected by both the airflow rate and the concentration of the froth stabilizer [60-62]. Equation (4) is the standard method originally proposed by Bikerman [61].

$$\Sigma = \frac{V_f}{Q} = \frac{H_{max} A}{Q} \quad (4)$$

Where are:

A - cross-sectional area of the vessel (cm^2),

Q - gas volumetric flow rate (L/min),

H_{max} - total froth height (cm),

V_f - froth volume (L),

Σ - dynamic froth stability (min).

3. Results and Discussion

3.1. Effect of frother structure

The molecular chain length is a crucial factor in determining the performance characteristics of frothers. In frothers with longer molecular chains, such as PEG 300, the molecules can more easily spread across the surface of the bubbles, forming protective layers that provide greater froth stability. This structure helps prevent the froth from collapsing quickly and makes it more resistant to environmental changes or agitation. In other words, longer chains can create a more organized structure on the surface of the bubbles, leading to increased stability. On the other hand, frothers like TEG, which have shorter molecular chains, are less capable of forming protective layers and stable structures. As a result, the foam produced by these frothers tends to be less stable and more sensitive to agitation and environmental conditions. Overall, the molecular chain length directly affects the froth's stability and its behavior under various conditions.

3.2. Effect of frother concentration on Frothability

The volume–air flow rate plots that were obtained for DF-250, PEG300, DPG, and TEG frothers procedure are shown in Fig 3. The steady-state froth height versus flow rate for the four different frothers is shown with varying frother concentrations. The figure clearly shows that the froth volume increases with increasing air flow rate and frother concentration for all the tested frothers. It may also be observed from the figure that DF-250 gives the highest froth volume, and DPG produces considerably low frothing. For the more powerful frothers, the volume–Air flow rate plots do not lend themselves to easy analysis. Therefore, determining DFI is very important. The retention time values were plotted against the corresponding concentration for the desired frothers and obtained from the slope of the linear portion of the dynamic frothability index graph. (Fig4). The DFI values for the tested frothers are given in Table 2. The order

of frothing power among the four frothers, in terms of the dynamic frothability index, is given below:

DF 250>PEG300>TEG>DPG

PEG 300 has a lower DFI than DF-250, which means it produces less froth, but what it does produce is more stable. DPG has not been able to provide long-term stability compared to other frothers due to the chemical characteristics of its structure that lead to faster froth degradation and TEG, which exhibits a behavior between the other two frothers.

In the evaluation of froth stability characteristics, a significant difference in the performance of frothers was observed. While frothers such as PEG300 produced relatively less foam, their more stable structure allowed for better froth retention under agitation. This may indicate the role of parameters such as the molecular chain length and the interaction between surfactants and bubbles. In contrast, frothers like DPG, due to their physicochemical properties, were unable to provide the required stability, and the froth they produced was mostly unstable.

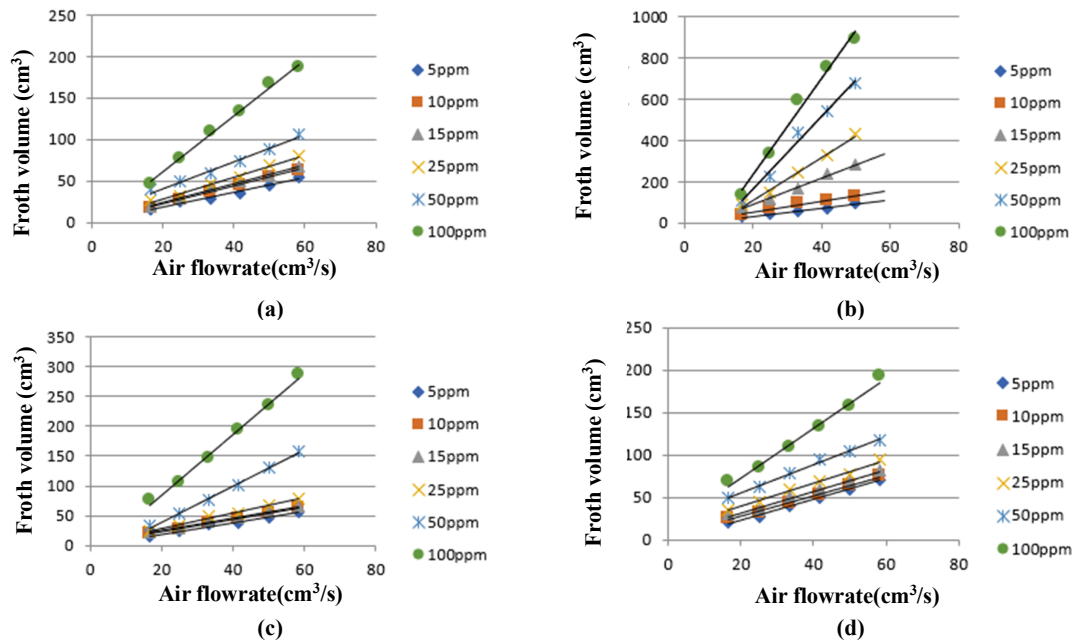


Figure 3. Effect of frother concentration on retention time: (a)TEG; (b) DF-250; (c) PEG300 and (d) DPG.

Table 2. Experimentally determined DFI values for the tested frother

Frother	DFI (s.dm ³ /mol)
Tetraethylene glycol	5117.7
PEG 300	13657
Dipropylene glycol	2544.9
DF-250	216906

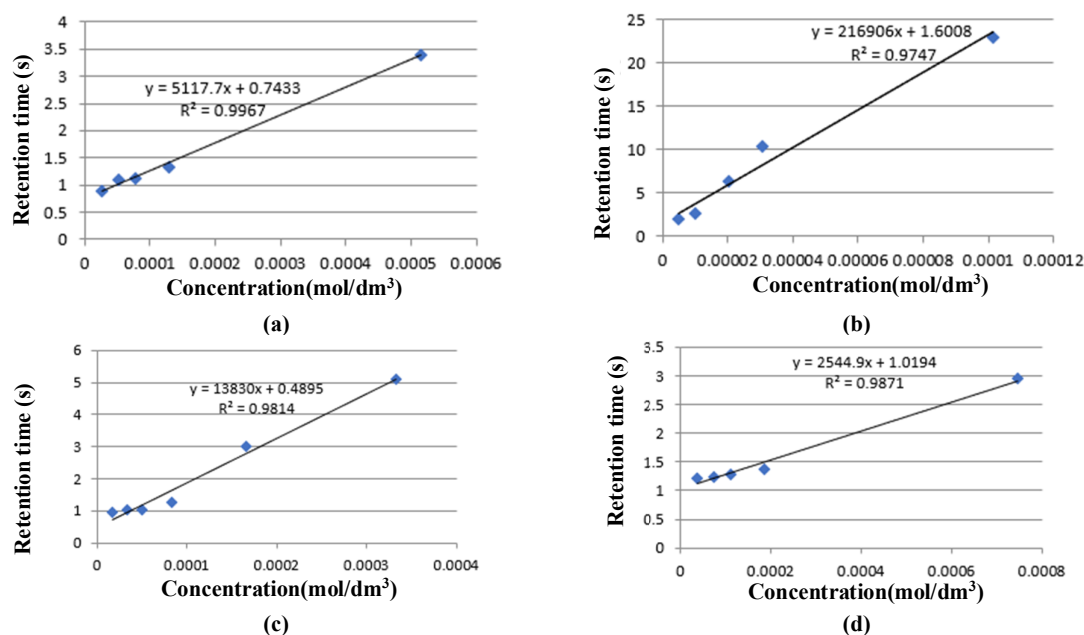


Figure 4. Graphical determination of DFI for TEG; DF-250; PEG300 and DPG Frothers

3.3. Effect of frother concentration on equilibrium froth volume

Figure 5 illustrates the relationship between equilibrium froth volume and frother concentration at various airflow rates. It is evident that increasing the concentration of frothers significantly affects both the volume and the stability of the foam. At low concentrations, frothers are not sufficiently dispersed in the medium, leading to weak interaction with bubbles and the formation of unstable froths. As concentration increases, these frothers interact more effectively with the air-liquid interface, forming more cohesive and elastic films around bubbles, which contributes to higher foam volume and longer retention time. However, at very high concentrations, excessive surfactant accumulation can increase the viscosity of the system and amplify turbulence under high airflow rates, leading to faster foam collapse. This suggests that there is an optimal concentration range beyond which the benefits of increased frother presence may reverse. Additionally, the interplay between airflow intensity and frother concentration becomes critical, as stronger shear forces at high aeration can disrupt even stabilized foams. Thus, both the physicochemical properties of the frother and the operational parameters such as airflow must be carefully balanced to maintain stable froth characteristics.

3.4. Effect of air flowrate on equilibrium froth volume

Figure 6 shows the relationship between the equilibrium volume of the froth and the airflow rate at four different frother concentrations. It can be seen that at low surfactant concentrations, the equilibrium froth volume generally increases when the air flow rate is increased. The dynamic stability factor (Σ) corresponding to the previous results is shown in Figure 7. DFS also showed similar results to the DFI. It is observed that the dynamic stability index initially increases with increasing aeration rate and then decreases due to turbulence at high aeration rates.

Furthermore, increased aeration may lead to the formation of unstable froths that collapse immediately after formation. This can lead to reduced dynamic froth stability at higher aeration rates. PEG 300 has a lower DFI than DF-250, which means it produces less froth, but what it does produce is more stable. DPG has not been able to provide long-term stability compared to other frothers due to the chemical characteristics of its structure that lead to faster froth degradation, and TEG, which exhibits a behavior between the other two frothers, is less stable than PEG300 due to its shorter molecular chain length.

In addition to the chemical and physical properties of each frother, the type of variable and parameter used also affects its performance. For example, in high aeration systems, frothers with more resistant structures, such as PEG 300, will perform better. In contrast, in systems with lower aeration, frothers with higher surface activity but lower stability may still provide acceptable performance.

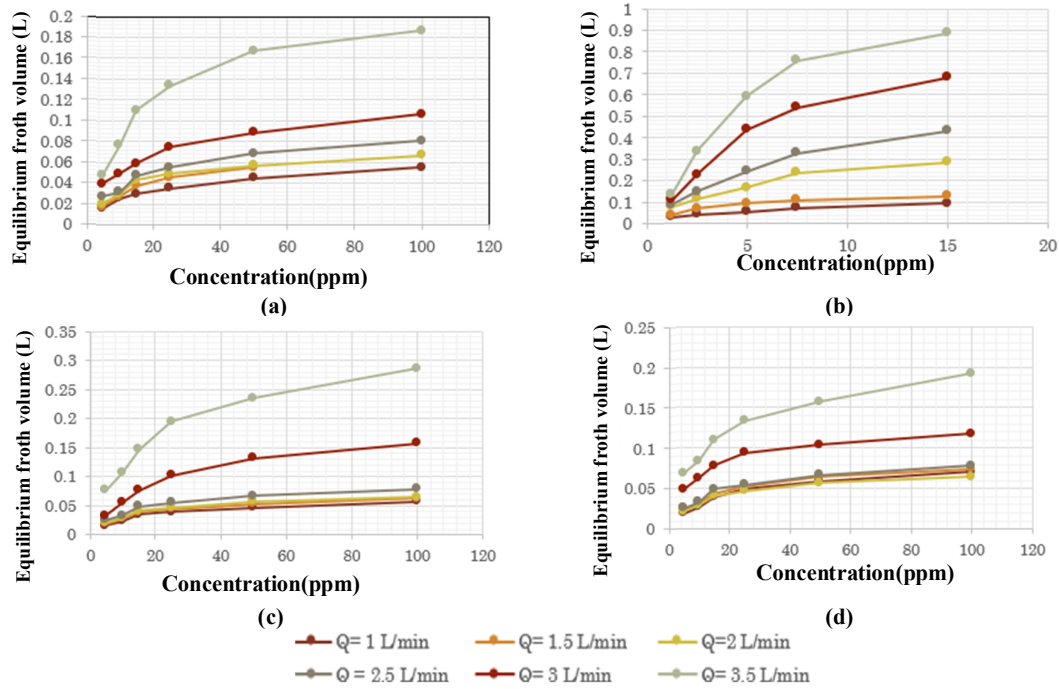


Figure 5. Equilibrium froth volume as function of frother concentration for different air flowrates: (a)TEG; (b) DF-250; (c) PEG300 and (d) DPG.

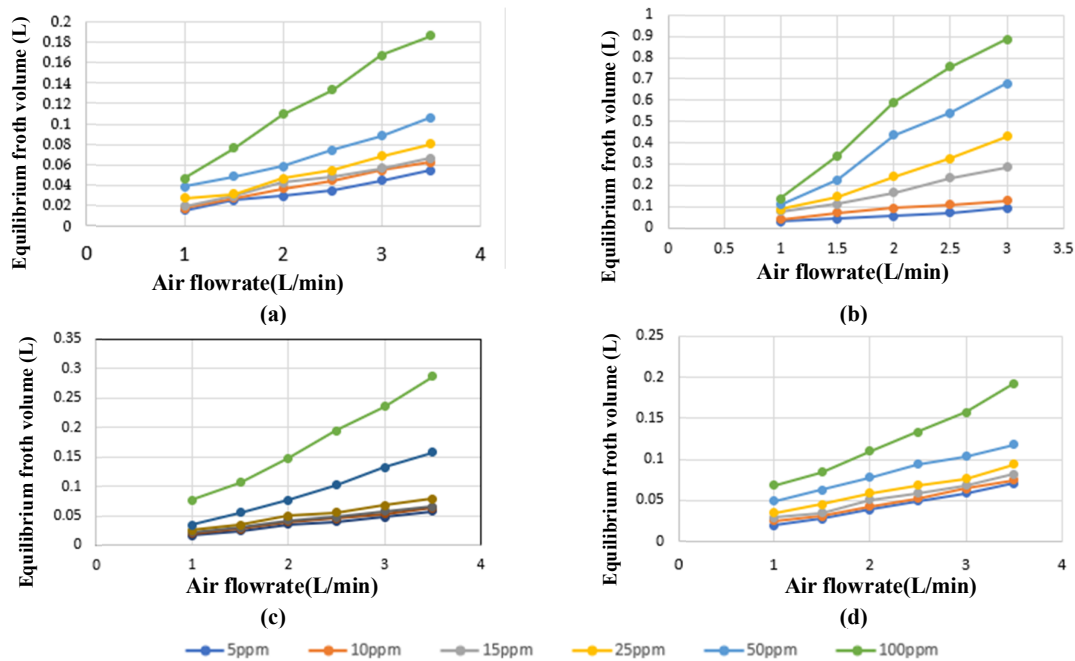


Figure 6. Equilibrium froth volume as function of air flowrate for different frother concentrations: (a)TEG; (b) DF-250; (c) PEG300 and (d) DPG.

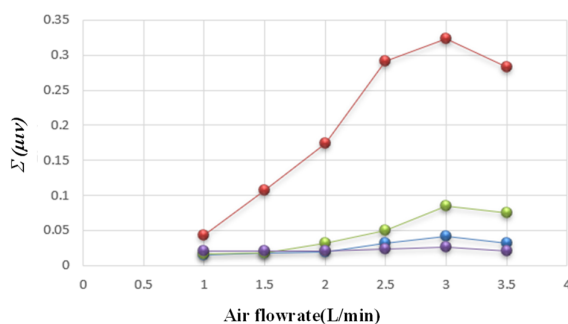


Figure 7. Dynamic stability index as function of air flowrate for TEG; DF-250; PEG300 and DPG frothers.

4. Conclusions

The effect of froth concentration and Air flow rate on froth height and retention time was investigated to characterize polyglycol-based froths. It is worth noting that these frothing properties are independent of bubble size. The frothers studied (PEG300, DPG, and TEG) were characterized based on the Hydrophilic-Lipophilic balance number, dynamic froth stability, and dynamic frothability of the frothers. DFI in this comparison showed that DF-250 showed significantly the highest froth production and stability. This could be due to its specific chemical structure, which produces stable froth and higher volume. In contrast, PEG300, despite its good frothing power, performed slightly worse than DF-250 in stability. This indicates the importance of molecular structure and chemical properties in froth stability. Based on the results of dynamic froth stability at low to medium air flow rates, good froth volume and stability can usually be achieved. However, at high, the froth volume may increase rapidly, but this increase in volume will be accompanied by a decrease in dynamic stability. The molecular structure of frothers and their chemical properties can also play an important role in maintaining sufficient stability against changes caused by increased air. This study showed that the molecular characteristics of three different frothers significantly affect their frothing power and dynamic froth stability. PEG300, with its long and linear structure, showed the highest frothing power and froth stability. On the other hand, DPG, with its branched structure, showed the lowest froth stability. TEG, with its medium-length linear structure, performs between the two in terms of frothing and stability. This study can help in the selection of polyglycol-based frothers for their efficiency in flotation of coarse and fine particles.

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انجمن مهندسی معدن ایران

مشخصه یابی کف سازهای مبتنی بر پلی گلیکول: بررسی پایداری دینامیکی کف و قابلیت کف سازی دینامیکی

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چکیده

ارزیابی عملکرد کف سازها از طریق شاخص های مختلف برای درک چگونگی تأثیر ساختار مولکولی بر عملکرد آنها و همچنین ارزیابی اثربخشی آنها در شناورسازی ذرات ریز و درشت بسیار مهم است. این مطالعه برای اولین بار رفتار کف سازی و پایداری کف پلی اتیلن گلیکول ۳۰۰ (PEG300)، دی پروپیلن گلیکول (DPG) و تتراتیلن گلیکول (TEG) را بررسی کرده و آنها را با کف سازهای مرسوم مانند Dow Froth-250 (DF-250) مقایسه می کند. برای ارزیابی عملکرد کف ساز، سرعت جریان هوا و غلظت کف ساز به عنوان متغیرهای عملیاتی اصلی مؤثر بر تشکیل کف و شاخص پایداری انتخاب شدند. در ابتدا، رفتار کف سازی معرف ها با استفاده از نمودار HLB-MW پیش بینی شد و سپس قدرت کف سازی کف سازهای مورد نظر با استفاده از شاخص های کف سازی دینامیکی و پایداری دینامیکی کف بررسی شد. نتایج نشان داد که PEG300 بالاترین شاخص کف سازی دینامیکی (۱۳۰۰۰ s.dm³/mol) و پایداری بالای کف را نشان می دهد که برای شناورسازی ذرات درشت مناسب است. در مقابل، DPG با شاخص کف دینامیکی ۲۵۰۰ s.dm³/mol کمترین قدرت کف سازی و پایداری کف را نشان داد. TEG با شاخص کف سازی متوسط ۵۰۰۰ s.dm³/mol، عملکرد متوسطی را در تولید کف و پایداری نشان داد. DF-250 با شاخص کف سازی فوق العاده بالا، از سایر عوامل پیشی گرفت و هم تولید کف و هم پایداری برتر را فراهم کرد. پایداری کف با استفاده از شاخص های پایداری کف دینامیکی و قابلیت کف سازی دینامیکی ارزیابی شد که بینش معناداری در مورد عملکرد کف ساز ارائه می دهد. نتایج همچنین نشان داد که هم سرعت جریان هوا و هم غلظت کف ساز تأثیر قابل توجهی بر شاخص کف سازی و پایداری دارند و غلظت های بالاتر عموماً پایداری کف را افزایش می دهند، به ویژه برای PEG300 و DF-250.

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