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A new method for determination of frothability of frothers using water recovery measurement

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ABSTRACT

A new method was introduced for frothing characterization of flotation frothers. The method uses water recovery to develop a new frothability index named water recovery index (WRI). This index was determined for some commercial frothers and the results were compared with dynamic frothability index (DFI). The results show that the water recovery index values follows the order of A-65 13016 s/mol > DF-250 6292.4 s/m > MIBC 1240 s/mol > Isoamyl alcohol 343.2 s/mol > Butanol 144.87 s/mol. It also shows that the DFI order is A-65 437,080 s.dm^3/mol > DF-250 197,271 s.dm^3/mol > MIBC 39,427 s.dm^3/mol > Isoamyl alcohol 10,517 s.dm^3/mol > Butanol 1977.3 s.dm^3/mol. The new method offers many advantages over conventional froth height measurement; the experimental set-up developed for water recovery measurement is more compact and is easy to use. Moreover, the special design of the set-up on the other hand, eliminates the wall effect of flotation container and increases the reproducibility of measurements.

Keywords: *Dynamic frothability index; Flotation frother; Froth; Froth stability; Reproducibility; Water recovery index*

1. Introduction

The importance of frothers in froth flotation is well known due to their role to asset the froth stability, reduce the coalescence of air bubbles in pulp and froth zones, and reduce the rising rate of bubbles from pulp zone to the froth phase [1]. Frothers are heteropolar compounds containing a non-polar water-repellent group, as well as a single polar water-avid group [2]. Frothers accumulate preferentially at the air-water interface such that the hydrophilic or polar groups are oriented into the water phase and the hydrophobic or non-polar hydrocarbon chain in the air phase [3,4]. However, in order to accept a surfactant as a flotation frother, some frothability characteristics must be taken into account, since a good frother has to achieve a delicate equilibrium between the balancing and non-persistency. That is, the frother should be able to produce a froth phase with a high volume to build a high mineral recovery and keep steady some bubble coalescence or collapse rate to guarantee a feasible concentrate grade [5].

Froth properties, i.e. structure and stability, play a key role in determining the flotation performance. Thus, the parameters affecting froth properties have to be properly investigated (e.g. [6-10]). Different methods have been developed for characterization of frothability of frothers in both two-phase (liquid/air) and three-phase (particles/water/air) systems.

In general, two types of tests have been proposed to characterize the frothability of frothers: static tests and dynamic tests. In static tests, a characteristic time is used to describe the froth stability (half-life time). It is defined based on the time spent for the froth to fall down half of its initial equilibrium height when the aeration is stopped [8]. Then the froth half-life time and the corresponding equilibrium froth height are used to evaluate frothability of the frother. A frother with strong frothability tends to produce a high and stable froth. However, there are several limitations in generalization of static frothability measurements for industrial applications [5]:

- Some frothers produce a low volume of froth, but they improve the flotation performance;
- Two frothers with the same static frothability may have different flotation performance, and;
- Static frothability measures are quantitative parameters, not inherent properties of a frother.

Dynamic tests use a frothing parameter known as dynamic frothability index (DFI) which is actually derived from static data, i.e. froth height and retention time. The latter is equal to the ratio of froth volume to gas flow rate. Then, DFI is defined as the limiting slope of retention time when the frother concentration approaches zero.

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Frothers with higher DFIs produce froths with higher volume and stability [11]. DFI is considered as an inherent property of each frother. DFI values for different frothing agents have been measured and widely applied by researchers [4,7,12-17]. Successful DFI measurement at plant scale is being reported by some investigators as well [18,19]. However, there are some problems that challenge the reliability of DFI data:

- The reproducibility of DFI measurements significantly decreases when testing powerful frothers such as DF-250. In such cases, due to having a very high stability, the rising froth tends to attach to the inner wall of the container (known as wall effect) and makes it difficult to read the exact height of the froth.
- DFI requires a constant value of retention time for a given frother and concentration, which is specifically obtained from the slope of froth volume versus gas flow rate linear curve. Usually many data points are required to indicate the linear section of volume-aeration rate curve. In addition, it has been observed that not all the frothers exhibit such a linear relationship. Thus it is inappropriate to arbitrarily select a linear section of the froth volume-aeration rate to represent the entire system [5,20].
- There are also some problems with measurement apparatus which will be discussed further in this paper.

Regardless of the method used for characterization of frothing properties, frothers with higher frothability increase the froth recovery. Stable froths have higher water content and thus the strong frothers will produce more water overflow. This occurs due to the frothers' ability to increase the froth recovery. Water recovery can therefore be considered as a frothability measure of a frother as applied by some investigator [13,21-25]. The aim of the present study is to develop a more reliable method for frothing characterization of flotation frothers and to introduce a new index based on the water recovery measurement as a measure of frothability. The new index was also compared with the dynamic frothability index.

2. Materials and Methods

2.1. Reagents

List of the frothers tested in this research is given in Table 1. The reagents used in the experiments were of industrial grade.

Table 1. List of frothers used in this research study.

Frother	Formula	Molecular weight (g/mol)	Dynamic frothability index (DFI) (s.dm³/mol)		Water recovery index (WRI) (s/mol)
			Measured	Reported	
Butanol	$CH_3(CH_2)_3OH$	74.12	1977.3	1339[a]	144.87
Isoamyl alcohol	$(CH_3)_2CHCH_2CH_2OH$	88.17	10517	-	343.2
MIBC	$(CH_3)_2CHCH_2CHOHCH_3$	102.17	39427	34000[b], 35020[c], 36991[a], 37000[d]	1240
DF-250	$CH_3(C_3H_6O)_4OH$	264.37	197271	208000[b]	6292.4
A-65	$H(C_3H_6O)_{6.5}OH$	395.61	437080	-	13016

[a] Cho and Laskowski [7]; [b] Laskowski [12]; [c] Gupta et al. [4]; [d] Melo and Laskowski [13]

2.2. Dynamic frothability index measurement

The DFI values for tested frothers were determined using a well-known procedure first introduced by Bikerman [26]. The dynamic frothing of frothers is measured in a column frothmeter. The foaming vessel used in this paper is a cylindrical column made of graduated glass with 5 cm inner diameter and 120 cm height which is fixed by a glass frit at the bottom. The fixed glass has a diameter of 45 mm and pore size of 40–100 µm. The flow rate of air is controlled via a rotameter calibrated for air in the range 1 to 10 L/min and a needle valve.

To start the test, the froth column was filled with 200 ml of the frother solution of known concentration and air was bubbled into the solution at predetermined flow rate to create froth. The DFI method requires determination of gas retention time in column as a function of gas flow rate and frother concentration. The measured value is the total height of the solution plus the foam phase. Therefore, the initial height of liquid in the column, H_i, was recorded before passing air into the column. When the froth height reaches the equilibrium, the total froth height (maximum height of froth), H_f, was recorded. The froth volume, which is the froth height (H) times the cross sectional area of the column, is measured to determine the frothability of the solutions of frothers at different concentration levels and aeration rates. The froth height, H, is the summation of froth and bubbles trapped in the liquid per unit cross sectional area of column, or the difference between total froth height, H_f, and initial liquid height, H_i [5]:

$$H = H_f - H_i \quad (1)$$

Each test was carried out twice and then the average froth height was reported. All experiments were conducted at a room temperature of 25°C.

2.3. Water recovery index measurement

Figure 1 shows the experimental set-up designed for water recovery measurements. The set-up is made of a graduated cylinder (1) with an inner diameter of 5 cm and a height of 35 cm, which was used as the flotation column. The column was put inside a 4 L beaker (2) to collect the froth overflowing the column. Air bubbling was conducted using a small cylindrical sinter (3) with a diameter of 2 cm and pore size of 40–100 µm. The sparging cylinder from one side was put 1.5 cm above the bottom of the column, and from the other side was connected to the air source through a thin tube. Since the forming froth continuously discharges during the aeration, a graduated separatory funnel (decanter) (4) was installed on top of the column as a supplementary water resource to maintain the solution-froth interface zone at a given level. However, it was very difficult to visually distinguish the interface level, especially for powerful frothers, since the swarm of bubbles occupied the entire column; therefore, a thin glass tube (5) was completely connected to the inner wall of the column so that the side that was placed inside the column was 0.5 cm above the bottom of the column, but 1 cm below the end of the sparger to prevent the entry of air bubbles into the tube. In addition, the indicator tube was chosen long enough to prevent the froth from draining, i.e. 7 cm above the column opening.

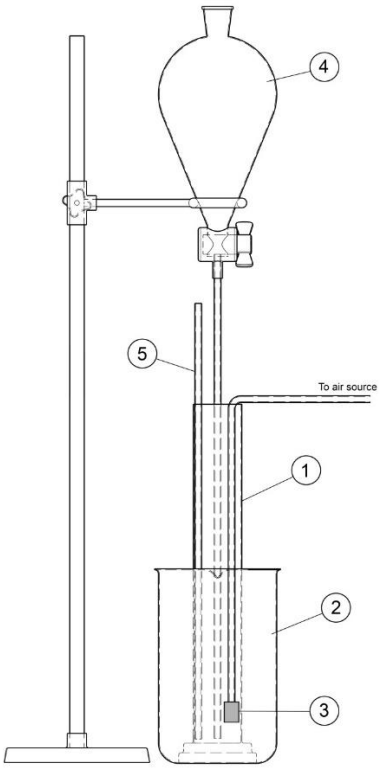

Figure 1. Schematic illustration of experimental set-up used for water recovery measurement.

The water recovery measurement for each frother was carried out as described below:

- 550 ml of solution with predetermined frother concentration was added to the graduated cylinder (column).
- The water tube from water funnel was put inside the column.
- The sparger was put inside the column and the air flow rate was set to the required value.
- The froth was allowed to overflow the column into the collecting beaker.
- The solution-froth interface zone level was visually checked and maintained at 550 ml level by manually adjusting the water funnel valve (Figure 2).
- Having completed the solution frothing, i.e. no further froth overflowed into the column, the water tube and sparger were detached.
- Then the volume of water discharged from the water funnel into the column was read.

Water recovery for each test was calculated using the following equation:

$$R_w = \frac{V_a}{550 + V_a} \times 100 \quad (2)$$

where R_w is the water recovery (%) and V_a is the volume of water (ml) added to the column to control the level of solution-froth interface zone.

where C is the molar concentration of frother. Finally, it can be graphically determined from the slope of the initial linear portion of the rt vs. C curve. Figure 3 shows the equilibrium froth height versus airflow rate for five different frothers with varying frother concentrations. The figure shows that the foam height increases as the air flow rate and the frother concentration for all tested frothers are increased. Figure 3 shows that froth height–aeration rate curves could be divided into two sections for all tested frothers but in different conditions. The curves show linear trend at lower air flow rate and a nonlinear one at higher flow rate for all frothers. Therefore, the measurement of retention time through slope method may not be adequate in describing these curves. For the frothers higher frothabilities (like A-65), the foam height–aeration rate plots do not allow to be easily analyzed even at low air flow rates. In such cases, retention time values could be measured through fitting the curves with linear regression technique [4]. Using the equilibrium data of tested frothers from Figure 3 and Equation (3), the froth retention time of the frothers at different concentrations was plotted. The DFI values were then measured according to Equation (4) and as an example, graphically shown in Figure 4 for MIBC. The dynamic frothability index measured for tested frothers is listed in Table 1 and compared with values reported in literature.

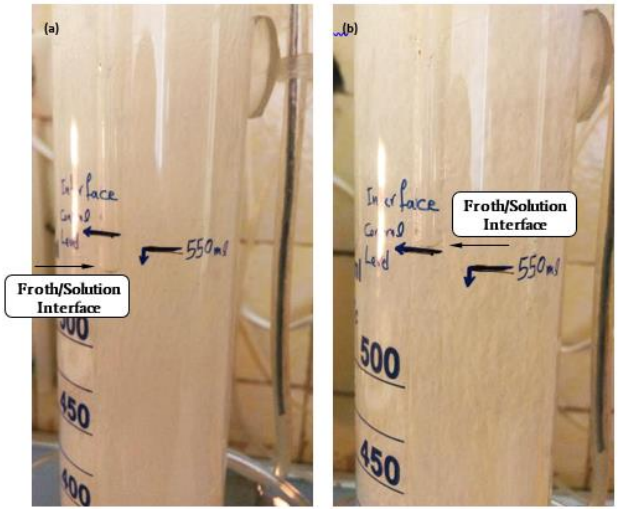

Figure 2. The level of solution-froth interface zone before (a) and after (b) adjustment (A-65, Concentration of 5 ppm, air flow rate of 3.5 l/min).

3. Results and Discussion

3.1. Froth stability and recovery measurements

The equations used to express the DFI as a function of gas flow rate and frother concentration are [11]:

$$rt = \frac{\Delta V}{\Delta Q} \quad (3)$$

where rt is the retention time (s), V is the total gas volume in the system (cm^3), and Q is the volumetric gas flow rate (cm^3/s). Then, the dynamic frothability index (DFI) is calculated from:

$$DFI = \left(\frac{\partial rt}{\partial C}\right)_{C \to 0} \quad (4)$$

where C is the molar concentration of frother. Finally, it can be graphically determined from the slope of the initial linear portion of the rt vs. C curve. Figure 3 shows the equilibrium froth height versus airflow rate for five different frothers with varying frother concentrations. The figure shows that the foam height increases as the air flow rate and the frother concentration for all tested frothers are increased. Figure 3 shows that froth height–aeration rate curves could be divided into two sections for all tested frothers but in different conditions. The curves show linear trend at lower air flow rate and a nonlinear one at higher flow rate for all frothers. Therefore, the measurement of retention time through slope method may not be adequate in describing these curves. For the frothers higher frothabilities (like A-65), the foam height–aeration rate plots do not allow to be easily analyzed even at low air flow rates. In such cases, retention time values could be measured through fitting the curves with linear regression technique [4]. Using the equilibrium data of tested frothers from Figure 3 and Equation (3), the froth retention time of the frothers at different concentrations was plotted. The DFI values were then measured according to Equation (4) and as an example, graphically shown in Figure 4 for MIBC. The

dynamic frothability index measured for tested frothers is listed in Table 1 and compared with values reported in literature.

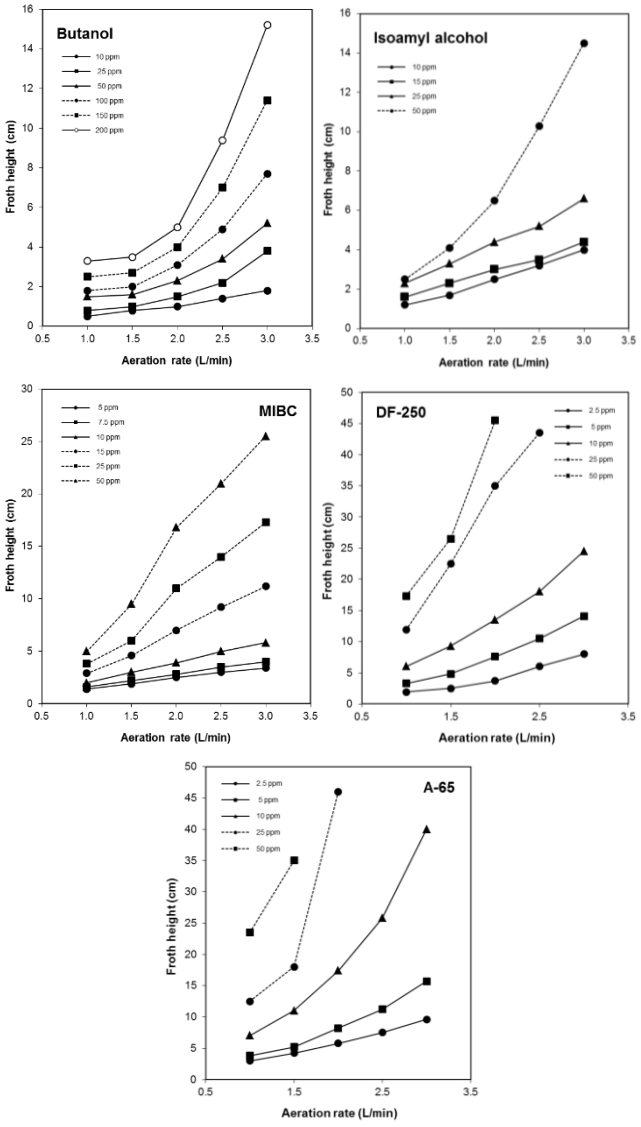

Figure 3. Froth height as a function of air flow rate and frother concentration for tested frothers.

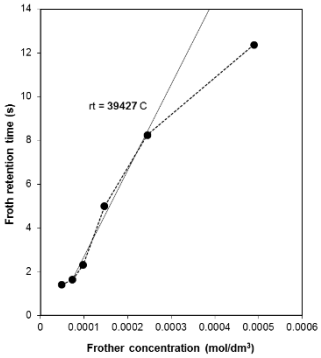

Figure 4. Graphical determination of DFI value for MIBC.

The water recovery values versus air flow rate for tested frothers are shown in Figure 5. After conducting the initial tests, the concentration values for each frother were selected based on their frothing capacity. As seen in the figure, water recovery-air flow rate curves show the same trend to the froth height-air flow rate. It is obvious that the curves follow a linear trend at lower air flow rates and a nonlinear one at higher flow rates. However, there is a difference: as air flow rate increases, the water recovery-air flow rate curve approaches to a horizontal line similar to the flotation kinetics curve, whereas the froth height-air flow rate shows an increasing trend. In DFI test, the froth grows to an equilibrium height. Since the froth column does not discharge, thus there is a dynamic equilibrium between the rate at which the frothers enter into the froth zone through rising bubbles and the rate at which the frothers drain back into the solution due to colliding and bursting bubbles into the froth zone.

In contrast, in water recovery tests, froth is allowed to be discharged continuously, and therefore, the solution gradually becomes frother free. It was also observed for all frothers that at low aeration rates, approx. below 3 L/min, the rate of water recovery is negligible. As the water recovery experiments were described, for each test 550 ml of solution was added to the column of 35 cm high. This volume of solution occupies about 28 cm of the container's height, and therefore, about 7 cm remains for producing the froth phase, i.e. the distance from the solution-froth interface to the opening of column. As it is shown in Figure 3, for tested concentrations on the froth height-air flow rate curves, the height of froth formed in frothmeter column for all frothers is smaller than 7 cm. In these cases, the air flow rate is lower than 3 L/min. So, it could be expected that at low aeration rates the froth does not reach the top of the column to overflow. There are two ways to help overflowing the froth; first, more volume of test solution can be added to the column to increase the water solution-froth interface. Initial experimental runs showed that the turbulence in the froth zone results in water splashing due to bubbly regime. Another way is to use the solutions with higher frother concentration. This is an alternative way at low aeration rates, because in some cases, the volume of the froth formed at high aeration rates is very high that the water from the collecting beaker is consumed to maintain the interface level overflows. In addition, as shown in Figure 5, the unusual trends appear at high frother concentrations so that the curves discrepancy is neglected. The main reason can be attributed to the fact that the frother concentration approaches to its critical micelle concentration (CMC). In MIBC case, for instance, CMC is about 100 ppm; therefore, as the frother concentration increase to CMC, the water recovery values approach to constant values.

3.2. Development of new frothability index

At constant aeration rate and froth height, the recovery of water depends on the stability of the froth phase. The stability of the froth phase is determined by the stability of liquid lamellae between gas bubbles, which in turn affects the froth water content. Therefore, the water recovery of the concentrate can be used as a parameter to evaluate the stability of the froth zone [27-29]. Since the tested frothers have different frothing capacities, various concentrations were used for water recovery measurement. Therefore, it was difficult to compare the frothability of the tested frother using only the water recovery curves shown in Figure 5. In order to evaluate frothability of the frothers using water recovery, an approach similar to DFI calculation was used, but the froth volume was replaced with the fraction of water recovered from the column. Then a new ratio parameter was defined:

$$M_{\mathrm{w}} = \frac{\Delta R_{\mathrm{w}}}{\Delta Q} \tag{5}$$

where M_{w} is a ratio parameter (s/cm^3), R_{w} is the water recovery fraction, and Q is the volumetric gas flow rate (cm^3/s). Instead of percent scale, the water recovery fraction was used to avoid burdensome calculations. Then, a frothability index, so called the water recovery index was calculated as follows:

$$WRI = \left(\frac{\partial M_{\mathrm{w}}}{\partial C}\right)_{C\to 0} \tag{6}$$

where WRI is the water recovery index (s/mol) and C is the molar concentration of frother. WRI can be graphically determined from the slope of the initial linear portion of the M_{w} vs. C curve. Calculation of WRI for MIBC is graphically shown in Figure 6. The water recovery

indices for other tested frothers were calculated and are given in Table 1. As shown in Table 1, WRI values are significantly smaller in magnitude than DFI values and this is an advantage when reporting and comparing data. It is interesting to note that both indices for all frothers show similar magnitude relations. For example, DFI value of DF-250 is about five times the MIBC DFI as can be observed for WRI values. This relation shows that the water recovery index can be considered as an inherent measure for every individual frother as is the dynamic frothability index.

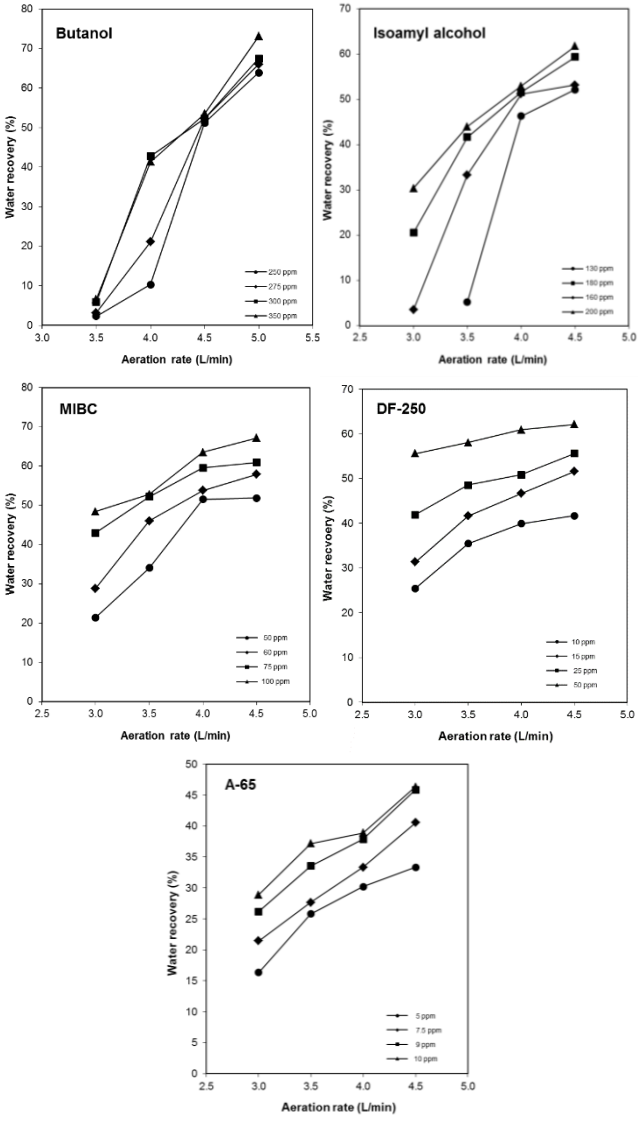

Figure 5. Water recovery as a function of air flow rate and frother concentration for tested frothers.

where WRI is the water recovery index (s/mol) and C is the molar concentration of frother. WRI can be graphically determined from the slope of the initial linear portion of the M_w vs. C curve. Calculation of WRI for MIBC is graphically shown in Figure 6. The water recovery indices for other tested frothers were calculated and are given in Table 1. As shown in Table 1, WRI values are significantly smaller in magnitude than DFI values and this is an advantage when reporting and comparing data. It is interesting to note that both indices for all frothers show similar magnitude relations. For example, DFI value of DF-250 is about five times the MIBC DFI as can be observed for WRI values. This relation shows that the water recovery index can be considered as an inherent measure for every individual frother as is the dynamic frothability index.

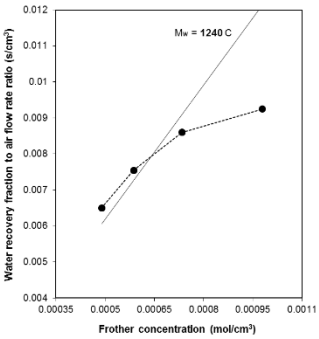

Figure 6. Graphical determination of WRI value for MIBC.

As mentioned earlier, reproducibility of DFI measurements significantly decreases while testing powerful frothers like DF-250 and A-65. In such cases, due to its high stability, the rising froth tends to stick to inner wall of the container, and then, it is divided into separate pieces (Figure 7). In some cases, it was observed that the air flow brings these pieces to the top of the column and finally discharges out of the container. One way to resolve this problem is to use the solutions with low frother concentrations. However, it may be necessary to increase the air flow rate to produce a froth column with adequate height. Increasing the aeration rate can negatively affect the froth stability by bursting the froth.

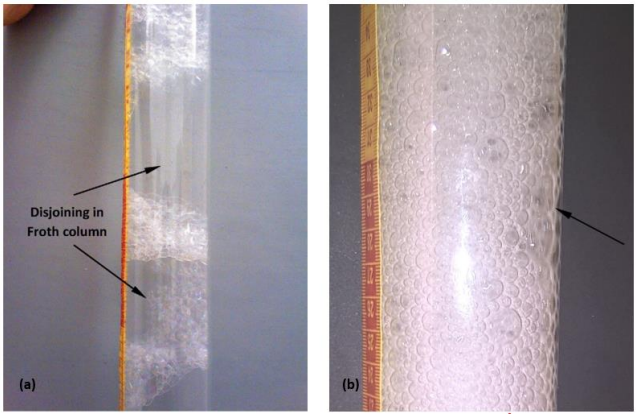

Figure 7. Disjoining in froth column produced by a) rhamnolipid (DFI 540,000 s.dm³/mol) [30] and b) DF-250 (DFI 197,271 s.dm³/mol, in this study).

There are also several problems with the frothmeter column. Different types of columns have been used by investigators as listed in Table 2. The container that effect DFI measurement are separated into three categories. The first category refers to the column size, i.e. height and diameter. Long columns are difficult to use because they should be carefully washed before each experiment. Moreover, they should be placed on the ground to easily add the frother solution. In this case, one has to bend to read the froth height. Experimental set-ups used for DFI and WRI measurement are shown in Figure 8. As seen, WRI apparatus is more compact which makes it easy and safe to work with. Authors have used containers with different diameters. It is shown that for a specific liquid volume fraction the froth drainage rate increases monotonically as the diameter is decreased. This trend can be attributed to the increasing wall films drainage compared to the interstitial films drainage. Although it is proved that the smaller containers increase the rigidity of the froth by limiting the elasticity of Plateau channels [31,32]. The second category is the container shape. Since most lab studies utilized cylindrical containers with circular cross section, the effect of column shape can be neglected.

The third category refers to the wettability of the container walls. As given in Table 2, most researchers used glass column in order to facilitate visual observations. Clean glass columns are fully wettable leading to hydrophilic drainage. Experience has shown that hydrophilic drainage over clean glass substrates gives good reproducibility of the experiments [32]. However, the wettability of glass may change with

time if it is not properly cleaned. This occurs when the surface active molecules adsorb on the glass surface. Some investigators have employed polymeric columns (Plexiglas or Perspex) for safety considerations. Polymeric materials are hydrophobic with varying degree of wetting properties due to raw material, manufacturing, surface treatment, etc. Hydrophobic materials create a wall effect and the froth may stick to the sides. However, it is shown that the differences between hydrophobic and hydrophilic walls disappear at larger column diameters (approx. larger than 40 mm) and any wall effects would be expected to diminish [17,32]. In practice, considerations have to be taken into account in measuring the water recovery index to eliminate the problems related to the froth stability and wall effect. Water-froth interface was kept as high as possible and the froth was continuously discharged so that the contact time between froth and column wall was negligible. Therefore, the hydrophobicity of column wall is not a concern and one can employ any type of materials for making the column. In addition, the length of WRI column is about three times shorter than DFI column which makes it easier and safer to use.

Table 2. Froth columns used for DFI measurement by different researchers.

Column type	Height (cm)	Inner diameter (cm)	Solution volume (ml)	Sinter porosity (µm)	Reference
Glass	92	4.5	-	-	Cho and Laskowski [7]
Glass	50	2.5	15	-	Gupta et al. [4]
Plexiglas	150	5.1	2000	40-60	Xia and Peng [5]
Glass	120	4.5	500	-	Schreithofer et al. [15]
Glass	60	5	200	40-100	Khoshdast et al. [16]
Glass	101	4.5	500	40-100	Castro et al. [11]
Plexiglas	150	8	-	-	Dey et al. [10]
Glass	100	4	150	90-150	Bournival et al. [9]
Perspex	100	9.6	2350*	40-100	McFadzean et al. [17]
Glass	120	5	200	40-100	In this paper

* as slurry

4. Conclusion

Both experimental and industrial investigations show that the water recovery can be reliably used as a measure for characterization of the flotation froths. Since frothers are of key importance in forming froths and determining their structure and stability, water recovery can be used as a measure for frothing characterization of flotation frothers. The new method introduced in this paper confirms the applicability of water recovery as a frothing indicator. Experimental results showed that the water recovery index gives similar results to the dynamic frothability index which is commonly used for characterization of flotation frothers. Although the new method has some limitations, it was shown that it can be superior to the conventional froth height measurement method. This superiority can be considered from safety, application and reproducibility point of view. The water recovery index seems to be applicable in industrial environments since it is easy to measure. However, further studies are required to develop the new method for industrial applications.

Figure 8. Comparison between dimensions of column frothmeter (a) and set-up used for water recovery measurement in this research study (b).

4.1. Reproducibility study

A reproducibility analysis was conducted to evaluate the reliability of obtained results from both WRI and DFI methods. As discussed earlier, each test was replicated twice. Therefore, a third series of tests with similar conditions to the first series were run and the WRI and DFI values for three experimental sets were calculated separately. Afterwards, the standard deviation of each set was calculated and applied for comparing the reproducibility of the methods. The results are listed in Table 3. As seen, the standard deviation values for WRI measurement are smaller than DFI data. This confirms that WRI method can present more reliable results even after multiple replications. In addition, the results show that the standard deviation values for both methods increases when more powerful frothers are used. That is the reproducibility of measurements decreases in presence of powerful frothers as denoted by Cho and Laskowski [7]. However, the reproducibility of measurements using such frothers increases when WRI method is applied. It should be noted that DFI and WRI values reported in previous sections were calculated using the mean values of froth height and water recovery, respectively. These values were calculated instead of the average value of final DFI and WRI for each individual measurement.

Table 3. Reproducibility analysis of WRI and DFI measurements.

Run	Butanol		Isoamyl alcohol		MIBC		DF-250		A-65	
	DFI	WRI	DFI	WRI	DFI	WRI	DFI	WRI	DFI	WRI
Rplt. 1	1907.3	142.14	10717	356.7	38427	1187.5	192001	5718	457401	12144
Rplt. 2	2044	132.9	9968.1	330.3	39014.3	1206.6	210666	6108.6	380991	14968
Rplt. 3	2001.1	140.15	9609	344.9	34988	1280.6	179210.7	6468.1	494317	12438
Mean	1984.13	138.40	10098.03	343.97	37476.43	1224.90	193959.23	6098.23	444236.33	13183.33
S.D. (%)	3.52	3.51	5.60	3.84	5.80	4.01	8.16	6.15	13.01	11.78

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