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The application of filterability as a parameter to evaluate the biological sludge quality in an MBR treating refinery effluent

Aline Ribeiro Alkmim[a], Paula Rocha da Costa[a], Míriam Cristina Santos Amaral[a,*], Luzia Sergina França Neta[b], Bárbara Caroline Ricci[a], Sílvia Maria Alves Corrêa Oliveira[a], Ana Cláudia Cerqueira[c], Vânia Maria Junqueira Santiago[c]

[a]*Department of Sanitary and Environmental Engineering, Federal University of Minas Gerais, P.O. Box 1294, 30270-901 Belo Horizonte, MG, Brazil, Tel. +55 31 3409 3669; Fax: +55 31 3409 1879; email: miriam@desa.ufmg.br (M.C.S. Amaral)*
[b]*Chemistry Department, Federal Center of Technological Education of Minas Gerais, 30421-169 Belo Horizonte, MG, Brazil*
[c]*Research and Development Center of PETROBRAS S.A/CENPES, P.O. Box 68505, 21941-915 Rio de Janeiro, RJ, Brazil*

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ABSTRACT

Membrane bioreactors (MBR) have been extensively employed at industrial effluent treatment plants; however the membrane fouling has restrained its more extensive application. Filterability is an important parameter to evaluate sludge properties and the potentiality of membrane fouling in MBR, nevertheless the lack of standardization of its assessment method has made it difficult to understand its actual role on MBR performance and compare its results. In this context, this work aims to compare three filterability assessment methods described in the literature (TTF, FT, and SFI) regarding its capability to sense sludge quality variation and reproducibility, and evaluate the application of this parameter as a tool to monitor and control fouling in MBR treating petroleum refinery effluents. This study showed that, among the methods evaluated, time to filter was the most effective to assess the filterability, both in terms of its capability to sense sludge quality variation and reproducibility. The results have also shown that filterability is directly related to the membrane fouling potential, and can be used as a tool to monitor and control fouling process in MBR. Significant filterability correlations among total colloidal organic carbon (colloidal COT), extracellular polymeric substances, and floc average size were found, for which Spearman coefficients determined were 0.71, −0.63, and −0.63, respectively. Therefore, filterability has the potential to forecast and control fouling so that its use, together with other parameters, allows for process optimization and reduction of power consumption, while increasing the membrane lifetime.

Keywords: Filterability; Membrane bioreactor; Fouling; Refinery effluent treatment

*Corresponding author.

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1. Introduction

Membrane bioreactors (MBR) are effluent treatment systems that include separation processes performed by membranes and biological processes [1]. Although membrane fouling remains as a disadvantage of this technology, the smaller footprint and the better product quality make MBR more effective than other biological wastewater treatment methods such as, for example, the conventional activated sludge method [2].

Membrane fouling directly affects the permeate flux, besides increasing the transmembrane pressure system, which demands for higher power consumption, higher cleaning frequency, smaller membrane lifetime, and therefore higher operational cost. Membrane fouling is influenced by a series of factors related to the feed, membrane and operating conditions, and it is caused by the deposition of mixed liquor suspended solids (MLSS) over internal and external membrane structures due to interactions between sludge compounds and the membrane [2].

Membrane permeability is the most important parameter used to monitor MBR process. However, Gil et al. [3] stated that results only based on this parameter allows for inaccurate conclusions about the process as it will not provide specific information on what factors have actually been determining.

Many studies have tried to relate the permeability decline caused by fouling to the sludge properties. For example, Chang et al. [4], Judd [1] and Drews [5] presented a review regarding sludge parameters—floc size, MLSS, extracellular polymeric substances (EPS), soluble extracellular polymeric substances (SMP), among others, and how they are related to MBR fouling potential. As discussed in this work, due to the biologic system complexity, the conclusions found have been often controversial, and therefore they cannot be directly transferred to other systems.

Biologic sludge from MBR shows different compositions and characteristics. One of these characteristics is its filterability, which stands for the resistance placed by the fluid to pass through a permeable/porous like membrane, indicating the propensity for membrane fouling caused by biologic sludge. Therefore MBR fouling is a process, and filterability is a specific characteristic of the sludge.

According to Rosenberger and Kraume [6], the filterability is directly influenced by the sludge properties such as concentration of suspended solids, temperature, viscosity, distribution of particle sizes, and concentration of univalent and bivalent cations. Thus, the study of this parameter associated with other sludge properties may be used as a tool to control and prevent membrane fouling.

The effects of sludge properties have been often controversial in different studies. Lee et al. [7] showed that due to the dynamic layer formation on the membrane surface, high suspended solid concentrations improve the system filterability, while Nagaoka et al. [8] found a negative effect caused by suspended solids due to their high viscosity that could affect filtering process hydrodynamic.

Mikkelsen and Keiding [9] found that sludge containing more fractions of EPS tend to form more flocs, improving the filterability. Then again, Kim et al. [10] and Nagaoka et al. [8] reported that filterability decreases with increasing EPS concentration. Houghton et al. [11], pointed out an optimal EPS range to improve sludge filterability.

Studies based on sludge disintegration by using ultrasound showed that filterability decreases considerably as suspended particle sizes decrease [12]. Nevertheless, experiments carried out Sun et al. [13] showed that ozonization of biopolymeric agglomerates by reducing their sizes, would improve their filterability.

Filterability discusses much about sludge properties, but not necessarily much about the system as the tests usually comprise only a simple filtration. It is not always the system geometry used for the test resembles MBR geometry, which may lead to some apparently controversial conclusions.

Many studies have shown the application of filterability tests to monitor and control incrustation on MBR [10,14–16]. However, different methods have been employed such as *Capillarity Suction Time* (*CST*) [17], *Time to Filter* (TTF) [18], *Filter Test* (FT) [17], S*ludge Filtration Index* (SFI) [19], and *Delft Filtration Characterization Method* (DFCm) [20]. The lack of filterability method standardization has impaired the understanding the influence of filterability on MBR performance, and so the comparison among results. Even though, sludge filterability is an important characteristic that provides valuable information on membrane bioreactor filtration processes, and as its determination is considerably easier to be carried out, it may be a suited parameter to monitor and control fouling process.

In this context, this work aims to evaluate the filterability test used as a tool to monitor and control membrane fouling in MBR. Furthermore, different methods were used to compare filterability measurement findings regarding their capability to sense sludge quality variation and their reproducibility, and as well correlate the most reproducible test results and sludge quality parameters.

2. Methodology

2.1. Selection of filterability methods

Periodically, over 184 d, samples of sludge of an MBR used to treat refinery effluent were collected, and the filterability was determined by using three different methods to compare them. The MBR evaluated comprised an external submerged ultrafiltration membrane bioreactor. The membrane was a PVDF-based (polyvinylidene difluoride) polymer that had the conformation of a hollow fiber, and had an average pore opening of 0.04 μm. Both, biologic and membrane tank have aeration systems.

Altogether, 32 sludge samplings were carried out, while the methods evaluated were TTF, FT, and SFI. All tests were triplicated for each of the samples. All samples were homogenized before performing the tests.

Filterability tests based on TTF were performed by using the assembly schematically shown in Fig. 1(a). The assembly had a 100 mL graduate beaker, a 1,000 mL Büchner flask, Buchner funnel with a diameter of 9 cm, and e filter paper (Whatman glass microfiber 934-AH, φ 90 mm, pore size retention 1.5 μm). The filtering procedures were performed by using a vacuum pump that supplied a pressure of nearly 51 kPa. To carry out the trial, 200 mL of sludge were used. The time required to filter a 100 mL was recorded. The filterability value based on this method is given by the Eq. (1):

$$\mathrm{TTF} = \frac{\text{Filtration time}}{100\,\text{mL of filtrate}} \quad (1)$$

For filterability tests performed by the Filter Test method (FT), it used an assembly as shown in Fig. 1(b). For these trials, it used a Whatman quantitative filter paper, Grade 42, pore size retention 2.5 μm with a diameter of 185 mm folded up into eight folds, so that it would fit into the funnel as shown in Fig. 1(b). Fifty milliliters of biologic sludge sample were collected and filtered through a filter paper. The countdown started right after the beginning of the filtering process, and the volume was recorded after 5 min. The filterability value found by this method is given by the Eq. (2):

$$\mathrm{FT} = \frac{\text{Filtrate volume}}{5\,\text{min}} \quad (2)$$

For the filterability tests based on the Sludge Filtration Index method (SFI), it used an assembly as shown in Fig. 1(c). Five hundred milliliters of sludge sample were collected from the bioreactor being studied. The sample temperature was adjusted to 20˚C by a ice bath. The funnel trials, it was placed on a Macherey Nagel MN 85/70 BF filter paper with a pore size retention of 0.6 μm. The sludge sample was homogenized and poured into the filter, and it was then stirred up at 40 rpm as shown in Fig. 1(c). The countdown started with the permeate volume collected from the beaker reached 100 mL, and stopped when the volume of 150 mL was reached. The filterability value measured by this method was calculated based on the time recorded in seconds in relation to the suspended solid concentration (SS) as given by Eq. (3):

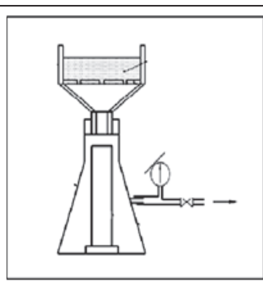
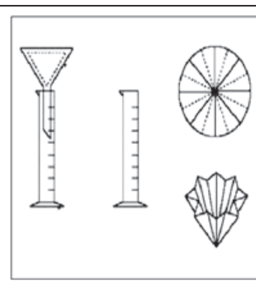
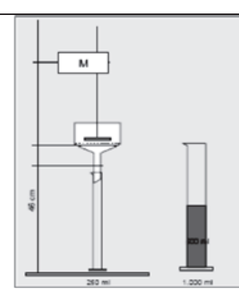
Time To Filter	Filter Test	Sludge Filtration Index
		
(a)	(b)	(c)
Source: THIEMIG [17] – Adapted	Source: THIEMIG [17] – Adapted	Source: THIEMIG [19]

Fig. 1. Assemblies used for sludge filterability tests.

$$\mathrm{SFI} = \frac{\Delta t}{\mathrm{SS}}\left[\frac{\mathrm{s}}{\%\mathrm{SS}}\right] \tag{3}$$

The findings of the tests were compared for sludge filterability variation sensing capability over the reproducibility monitoring time. Sludge quality variation sensing capability was evaluated by means of an analysis of the temporal behavior of the tests. In order to select the method that would allow for the highest reproducibility of the petroleum refinery sludge, the variation coefficient (VC) value sets resulted from the methods being tested were compared. To that, each test was triplicated for a determined sample, and the values found were analyzed by means of Dixon's *Q* test [21] to exclude the values deemed to be substantially different from the others. Then, the variation ratio was calculated from the median value of $\bar{x}$ and the standard deviation of s of the replicated tests as given by Eq. (4).

$$\mathrm{VC} = 100x\frac{s}{x} \tag{4}$$

As seen in Eq. (4), the VC represents the dispersion of a data-set in relation to its median value. So, when its calculated based on the replicated test results, the lower its value is, the lower data dispersion is, and higher it is its stability degree and, in such a case, higher the reproducibility degree is according to the test being studied. Besides, for being dimensionless, it allows for reproducibility comparison among the value sets of different measurement units, which explained its utilization to compare the filterability test results found in this work.

The substantiation of significant existing differences regarding reproducibility of filterability tests measured based on VC was evaluated by means of hypothetical test of multiple independent samples. To that, nonparametric tests were carried out, which do not depend on the frequency of the population being studied. So, to check for the significant existing differences among the median values of the variation coefficient, *Kruskal–Wallis test* was applied followed by a nonparametric test for multiple comparison among groups. All statistical analyses were performed by using STATISTICA 8.0 software.

2.2. The influence of the sludge filterability on membrane fouling process

To evaluate the influence of the sludge filterability on MBR fouling process, a MBR used to treat a petroleum refinery effluent was monitored, which was then associated with the permeability monitoring data. The sludge filterability was evaluated according to the TTF.

2.3. Influence of sludge properties on sludge filterability

The biologic sludge properties being evaluated as possible filterability influencers were concentration of mixed liquor volatile suspended solids (MLSSV) [18], SMP, and EPS were determined by thermal methods for EPS extractions introduced by Morgan et al. [22]; concentration of total colloidal organic carbon (colloidal TOC) was quantified by using TOC analyzer software (SHIMADZU TOC-VCPN) and determined according to the method proposed by Yang et al. [23], particle size distribution (laser scattering particle size distribution analyzer HORIBA—LA-950V2).

In order to verify the influence of biologic sludge properties on filterability, it was evaluated that the existence of a correlation coefficient among the parameters previously mentioned along with the respective filterability values found by applying the method that had shown higher reproducibility. To that, the Spearman correlation coefficient among the variables with further correlation significance analysis by means of the hypothesis test. STATISTICA 8.0 software was used to perform statistic tests.

3. Results and discussion

3.1. Selection of filterability methods

After carrying out Dixon's *Q* test, no data was deemed to be significantly different from the other values, and all were taken into account to compare methods regarding their sludge quality variation sensing capability and reproducibility. Fig. 2 shows the temporal filterability result series chart for the different tests being analyzed.

By comparing the results found, it was noticed that the FT method was unable to sense significant result differences sensed by using other methods being evaluated. It was observed that the filterability values found by applying the FT method showed an almost constant behavior, while the other tests showed significantly different values, particularly between the 70th and the 100th monitoring days. The finding indicated that, possibly in the case of the system being studied, SFI and TTF methods showed to be more sensitive to sludge quality variation compared to the FT.

In order to determine, which method would be the most reproducible to analyze the biologic sludge

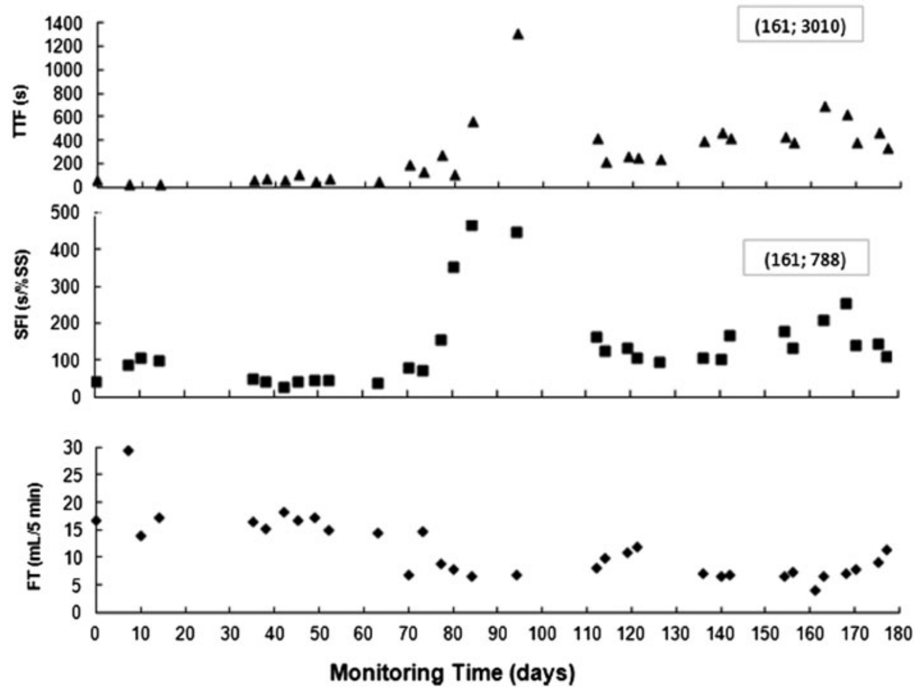


Fig. 2. Temporal comparison among the three filterability methods being analyzed. The highlighted values stand for the outliers.

obtained from an MBR that used to treat a petroleum refinery effluent, it performed a VC analysis of them. Kruskal–Wallis nonparametric hypothesis test was carried out to check whether the variation coefficients of the different filterability tests had median values significantly different at a significance level of 0.05. So it was found that there were significant differences among the median values, multiple nonparametric comparison procedures were adopted.

Such test allows for a simultaneous comparison between the VC data, aimed to identify which median values that taken two a time, would significantly differ from each other at the significance level of 0.05. Table 1 shows the multiple comparison test results, which were applied after verifying a significant existing difference among the groups found by applying Kruskal–Wallis test.

Table 1
Kruskal–Wallis statistic tests that showed significant differences among groups at a significance level of 5%

Test types	Result that showed significant differences
Filter test (FT)	Among TTFs
Time to filter (TTF)	Among all the tests
Sludge filtration index (SFI)	Among TTFs

By evaluating the results, it was observed that there were significant differences between the VC median values of the TTF method compared to the other methods, while there was no significant difference between the median values of the FT and SFI methods. Fig. 3 shows that the VC values of TTF method were significantly lower than the other test, which shows that such a test is the most stable of all the methods evaluated, and it is the one that features higher reproducibility to evaluate the filterability of biologic sludge used to treat petroleum refinery effluents.

In his study, Thiemig [19] compared the utilization of the three filterability methods in relation to their reproducibility. *SFI, FT, and Capilarity Suction Test* methods were compared for municipal sewage sludge treatment by means of analysis of (VC) tests. The method that had the worst result was the FT test, which confirmed the results found in the study performed. According to the author, this result is related to a small amount of sludge used for the test, which may cause higher variability of the results due to sampling errors.

Although the SFI method showed a sensing capability similar to the TTF method, for the biologic

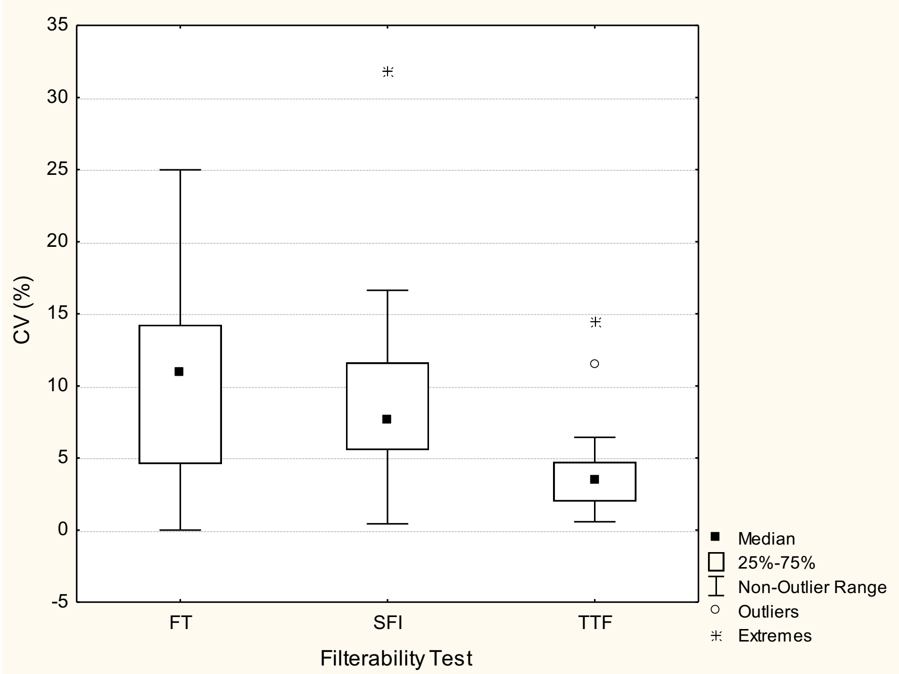


Fig. 3. Box-plot chart showing the range of variation coefficients of different filterability methods.

sludge being evaluated, its reproducibility was lower, which indicates that TTF method was the most proper for filterability evaluation of all them.

3.2. *Filterability as a parameter to evaluate the sludge fouling potential in an MBR*

In order to prove the quality of the reactional liquid has a direct influence on membrane fouling, MBR filterability and permeability TTF method results were evaluated as shown in Fig. 4.

As it may be seen in Fig. 4 that over the monitoring time the permeability decrement was followed by sludge filterability decrement (longer filtration time). So, it may be realized that filterability parameter, and as well other sludge parameters, can be used to forecast and monitor MBR fouling process. Besides it, being aware of the way other sludge parameters influence filterability may confirm the use of this parameter, which is easier to be used to monitor MBR performance.

3.3. *Influence of sludge characteristics in sludge filterability*

In order to evaluate the influences of sludge properties on filterability behavior, it was analyzed the possible existence of a correlation between these parameters. The following parameters were evaluated: colloidal TOC, SMP e EPS (characterized in terms of COD), MLSSV and particle size.

Fig. 5 shows the relationship between the sludge parameters and the filterability.

In order to evaluate the existence of such correlations, and to find out whether they are significant, the Spearman correlation coefficient (R) was determined, and the correlation significance test was carried out. The results found are shown in Table 2.

Based on the values obtained, it was found that there is a correlation between filterability determined by the TTF method and colloidal TOC parameters, EPS and Floc average size.

Regarding colloidal TOC parameter, the correlation coefficient found was about 0.7, which a statistically significant value at the significance level of 5%. That way, it was realized that colloidal TOC affects sludge filterability significantly so that the higher is the concentration, the longer the time required to TTF is, which indicates a sludge quality decrement.

Fan et al. [24] observed that colloidal TOC may be related to critical flux, a parameter used to control fouling process, and as well filterability, and defined as flux below which the membrane fouling would not take place [25]. An increase of colloidal TOC in the sludge causes a critical flux decrement. Such a phenomena is related to the increment of the potential fouling of biological sludge, which may also be related to filterability reduction. Therefore, colloidal TOC

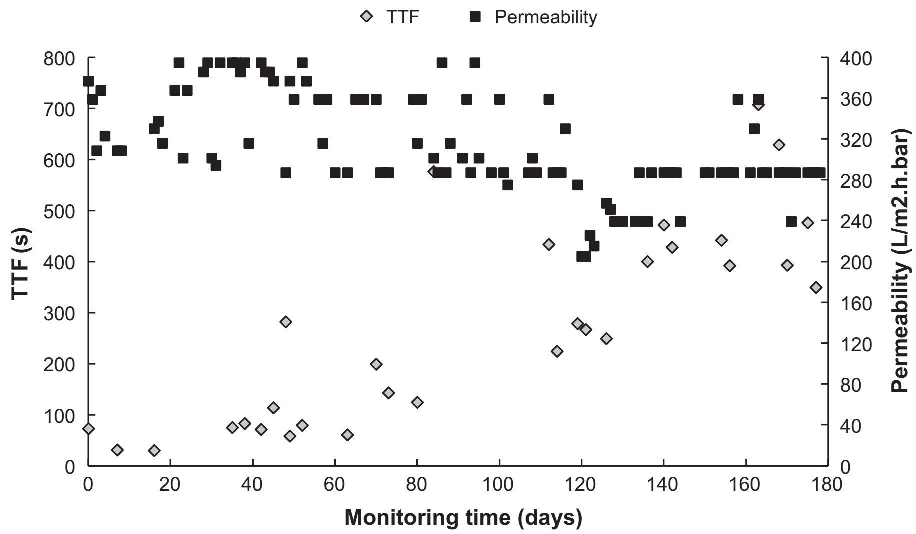


Fig. 4. Permeability and filterability profile by operation monitoring time.

likewise filterability may be suggested as prompt, reliable and easy to use indicators to evaluate the process behavior and control MBR membrane fouling process.

It was noticed that there was also a significant correlation between sludge filterability and particle size, and EPS concentration in terms of TOC, with correlation coefficients of about −0.63 in both cases. That way, it was found that by increasing EPS concentration and floc average size there was a sludge filterability improvement. In literature there is no consensus about the relation between the EPS concentration, floc size, and membrane fouling process. However, this answer may be associated with the contribution of EPS in the floc formation, which improves sludge filterability. Floc size increase favors higher filterability, form less dense and compact cakes, which have a little impact on the membrane fouling process [24]. According to Mikkelsen and Keiding [9], EPS parameter is the most important regarding the sludge structure and accounts for floc stabilization, and by increasing its concentration will make the flocs less sensitive to the shearing and there will be a smaller dispersion degree of them, which would bring forth a filterability improvement as it was found in the results presented.

Over the monitoring time, it was noticed the increment of concentration of solids in the system varying from about 8 to 9 g/L, while it was observed a sludge filterability decrease, which suggested that such parameters may be related (Fig. 5). However, no statistically significant correlation was found among them. Le-Clech et al. [26] studied the effect of MLSS concentration on the critical flux behavior. Regarding MLSS, no substantial difference was found on critical flux for a shift from 4 and 8 g/L. However, the greatest critic flux values obtained for 12 g/L were appointed as indicative of the formation of a protective gel layer on the membrane surface. Rosenberger et al. [27] stated that the general trend of MLSS increase on fouling process in municipal applications seemed to cause less incrustation at very low MLSS concentrations (<6 g/L), no impact at medium MLSS concentrations (8–12 g/L), and more incrustation at very high MLSS concentrations (>15 g/L).

That way, the effect of solids on fouling process is not clear mostly due to the complexity and variability of sludge biomasses. Thus, the variation of solids evaluated in this work has not been enough to determine the effect of the concentration of solids on filterability. No significant correlation between filterability and concentration of SMP was found in the system. According to Drews [5], two samples with the same SMP concentration may exhibit very different fouling behavior because its nature as a surrogate parameter means that individual components can be present in largely varying amounts and the physical-chemical environment (Ca concentration, pH, temperature) affects properties of SMP compounds which are relevant for fouling process such as size, shape, charge, gelling potential and hydrophobicity. Therefore the fouling potential measurement regarding SMP concentration could have multiple, complex and interacting influences that could not be assessed independently on each other in full-scale trials, and often are even more difficult to be separated in the lab.

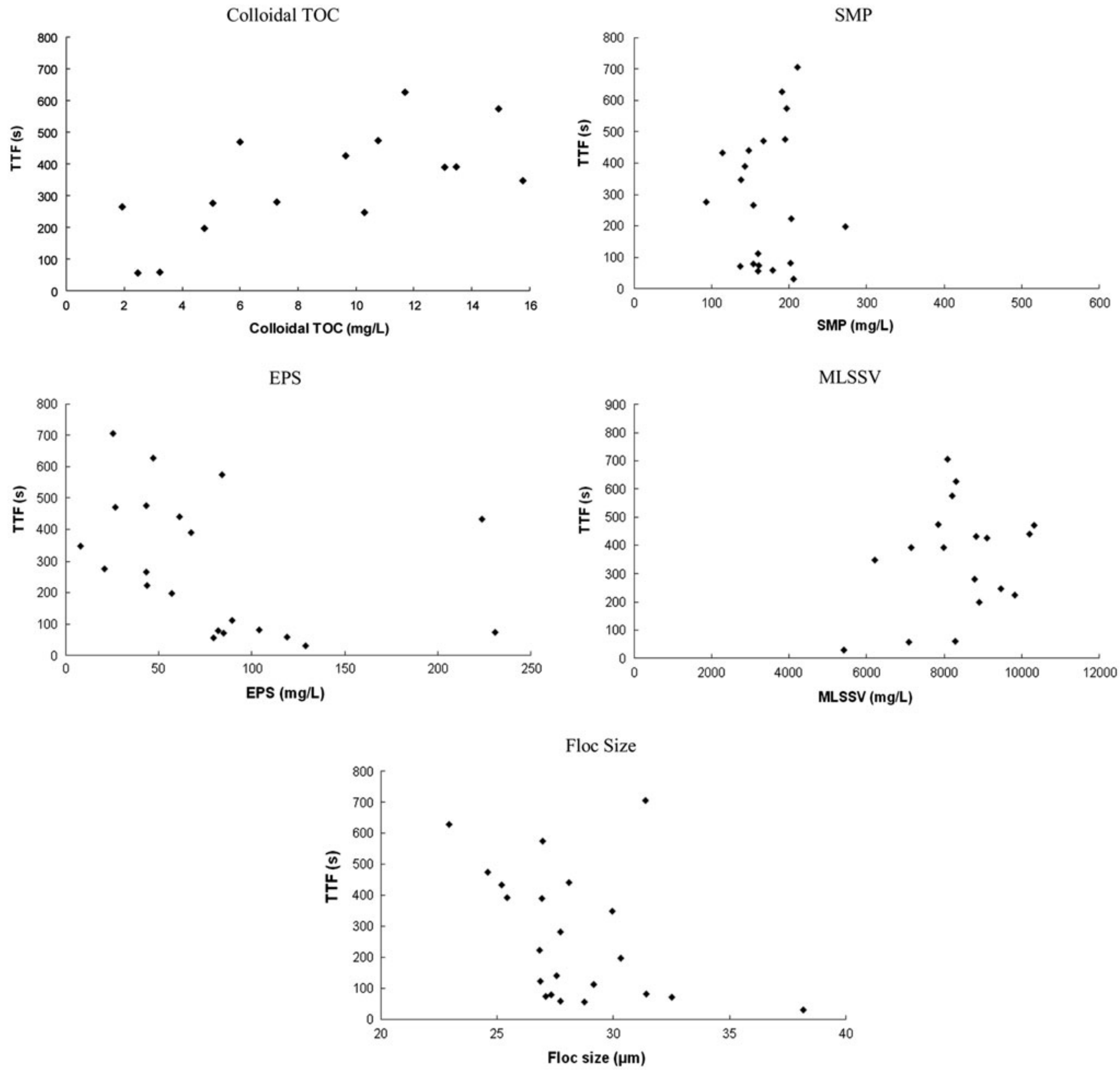


Fig. 5. Graphic of possible correlation between the parameters analyzed and sludge filterability found by TTF Test.

Based on the results obtained, it was noticed that filterability along with other sludge parameters may be used to forecast, monitor and control fouling process in the system being studied.

3.4. Filterability test towards sustainability in MBR operation

The stable long-term operation of membrane processes is imperative. Performance declination is both, inconvenient and costly. The major limitation is membrane fouling process that influences the selection of conditions for long-term sustainable operation [28]. That way, fouling process control allows for the process optimization, which cuts down power consumption, and extends membrane lifetime as maintaining membrane cleanliness demands further capital equipment (Capex) and operating costs (Opex) [29].

Therefore, filterability has the potential to forecast and control fouling process, and it is an easily determined parameter when compared to other factors reported in the literature. Sludge filterability

Table 2
Spearman correlation coefficient (*R*) values and correlation significance analysis *(The highlighted values correspond to the significant correlations.)*

Variable pair	Classification—Spearman correlation Marked correlations are significant = $p < 0.05$	
	Spearman (*R*) correlation coefficient	*p*-value
TTF and MLSSV	0.301471	0.239624
TTF and *colloidal TOC*	*0.714286*	*0.004104*
TTF and SMP	0.144415	0.543548
TTF and *EPS*	*−0.625564*	*0.003178*
TTF and *floc average size*	*−0.628120*	*0.001331*

decrement may be a warning to help implement some strategies to improve sludge quality such as adding permeability improvers, coagulants or adsorbents, effluent pretreatment, and other procedures carried out before a significant membrane fouling membrane takes place, which will reduce the chemical cleaning demand, and the MBR operational cost. As pointed out by Gil et al. [3], a good sludge filterability allows for a more sustainable and reliable MBR performance, and low power consumption.

4. Conclusions

The study showed that from the three filterability methods examined, namely TTF, FT and SFI, TTF test was the most efficient to measure filterability for its greater capability to sense sludge quality variations, and as well for its higher reproducibility. The FT was the one that attained the poorest outcome regarding the parameters being evaluated.

Based on the permeability monitoring results together with filterability, it was reasoned that filterability may be used as a tool to monitor and to help control MBR membrane fouling process, which demonstrates that reactional liquid quality has a direct influence on membrane surface fouling process.

By checking the filterability behavior for sludge properties, significant filterability correlations among colloidal TOC parameters, EPS and floc average size were found. So, the filterability has the potential to forecast and control the fouling process so that its use along with other parameter will allow for process optimization, lower power consumption and longer membrane lifetime, which will contribute to MBR sustainable operation.

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