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A Three Year Long Experience of Effective Fouling Inhibition by Threshold Flux Based Optimization Methods on a NF Membrane Module for Olive Mill Wastewater Treatment

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The operation of a spiral-wounded nanofiltration membrane module used continuously for three years in order to purify different pretreated olive mill wastewater streams will be discussed. The membrane module was for the first time used at the beginning of year 2006 on a batch pilot scale plant for critical flux studies and wastewater treatment in our laboratories.

The olive mill wastewater is a waste stream produced by the olive oil mill factories, characterized by very high organic matter load and polyphenols concentration. Without fouling inhibition at all, nanofiltration membranes will reach zero-flux conditions within days. This is not the case of this nanofiltration membrane module, which was successfully operated continuously for three years during our laboratory work. This result was reached by proper fouling inhibition control, relaying on both critical flux measurements and the development of an optimized operation method.

Although the critical flux theory was successfully applied to this system, it was not capable to explain the observed fouling behavior of the examined membrane system. The doubt to work on a membrane system that does not follow perfectly the critical flux laws grow throughout the years. In year 2011, Field et al. introduced the threshold flux concept as an extension to particular membrane systems treating real wastewater streams, and this latter theory fits to the observations made on olive mill wastewater.

In this work, a revision of previously obtained results in terms of critical flux will be performed, using the threshold flux theory as discussion basis. In the examined system, both critical and threshold points were found at 7-8 bar depending of the used feedstock and membrane condition. Moreover, it will be checked why the adopted "critical flux" approach was successful in inhibiting fouling for so many years despite it was not the correct approach. In a final step, both critical and threshold flux concepts will be merged within a single one, that is the boundary flux.

1. Introduction

As an indication of the popularity of membrane fouling problems, the growing evolution over the last 5 years has led to more than 3400 papers published in international journals satisfying this research subject (Scopus, 2012). Membrane fouling still remains nowadays one of the main challenges of the broad applied membrane technology, especially in liquid-liquid separation processes (Baker, 2004). The same behavior is not observed in other membrane system such as gas or vapor separation (Piemonte et al., 2011). Membrane fouling may lead to dramatically shorten the life time of membrane modules. For this reason, engineers design membrane processes with an excessive oversized capacity, up to 35% increasing both investment and operating costs (US Office of Water, 2005). This applies especially on wastewater purification processes (Lim et al., 2003).

Field et al. (1995) introduced the concept of critical flux for microfiltration, stating that there is a permeate flux below which fouling is not promptly observed. Afterwards, it was possible to identify critical flux values on ultrafiltration (”UF”) and nanofiltration (“NF”) membranes systems, too (Manttari et al., 2000). Nowadays, the

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critical flux concept is well accepted by both scientists and engineers as a powerful membrane process optimization tool (Bacchin et al., 2006).

The main drawback of this concept is that the determination of critical flux values cannot be theoretically predicted, but only experimentally measured by time consuming experiments. Moreover, different critical flux values can be measured on the same system, depending on various factors, such as hydrodynamics, temperature, feed stream composition and membrane surface characteristics (Vyas et al., 2002; Lipp et al., 1988; Zhou et al., 2009). Feed stream composition is the main responsible of variable critical flux values in case of agricultural wastewater stream treatment by membranes, since the entering feedstock quality is not constant during time. Moreover, the use of batch membrane processes in order to limit the amount of required membrane area and thus saving investment costs leads to sensible feedstock changes during operation. As a consequence, critical flux values never remain constant, which represent a major difficulty in fine-tuning optimal operating conditions.

In case of real waste water streams Le Clech et al. noticed that operations below the critical flux may not be sufficient in order to have zero fouling rates (2006). Therefore it appears that membrane systems treating real waste water streams do not exhibit a critical flux in strict way. To overcome this limitation in the definition of critical flux, in a recent paper, Field and Pearce introduced for the first time the concept of threshold flux (Field et al., 2011). Summarizing briefly the concept, the threshold flux is the flux that divides a low fouling region, characterized by a nearly constant rate of fouling, from a high fouling region, where flux dependant high fouling rates can be observed.

In the past years, before the concept of the threshold flux was introduced, the author published many papers on olive vegetation waste water (“OMWW”) purification by membranes, mainly ultrafiltration and nanofiltration, always determining critical fluxes (Stoller at al., 2006; Stoller et al., 2007; Stoller et al., 2010). Irreversible fouling arises quickly on the membranes due to the high concentration of pollutants when wastewater is purified without any pretreatment, and different pretreatment processes influences to variable extent the critical fluxes values (Stoller et al., 2009). Therefore, proper and optimal designed pretreatment processes on the given feedstock must be developed in order to maximize productivity and minimize fouling: this research objective will be referred from now on as the concept of pretreatment tailoring of membrane processes, and can be reached as an example by flocculation or photocatalysis (Sacco et al., 2012; De Caprariis et al., 2012; Stoller, 2011). The Authors observed in previous research works the change of fouling regime by using olive mill wastewater (Stoller, 2011; Stoller, 2008; Iaquinta et al., 2009; Ochando-Pulido et al., 2012; Stoller et al., 2006; Stoller et al., 2013).

In this work, previously measured critical flux data will be analyzed again by using the threshold flux theory as discussion basis, in order to check, and why in the past the adopted "critical flux" approach was capable to inhibit fouling for so many years. Moreover, the boundary flux concept will be introduced, merging both critical and threshold flux concepts into a single one.

2. Experimental

2.1 The wastewater stream

Olive mill wastewater (“OMWW”) is a heavy polluted heterogeneous liquid stream exiting the olive oil production process. It is characterized by an acid value (pH value equal to 4), very high COD value (up to 200 g l^{-1}), suspended solids and a high concentration of phenols (more than 300mgl^{-1}). A medium sized olive oil mill gives rise to around 10m3 day^{-1} of this wastewater, which represents a major threat the environment, a great cost for its disposal and a huge amount of potable water consumption. This wastewater has antimicrobial and phytotoxic properties, cannot be disposed for irrigation purpose and is resistant to biological degradation, thus biological treatment results difficult and up to today industrially not feasible. Moreover, irreversible fouling arises quickly on the membranes due to the high concentration of pollutants when the wastewater is separated without any pretreatment. The pretreatment processes includes flocculation and photocatalysis, widely used on wastewater streams (Di Palma et al.).

2.2 The lab scale plant

The used pilot plant is shown schematically and as photograph in Figure 1.
The plant consist of a 100 l feed tank FT1, in which the pretreated feedstock is carried. The volumetric pump P2 drive the wastewater stream over the spiral wounded nanofiltration membrane, by Osmonics, fitted in the housing M1, with an average flow rate equal to 600 l/h. This membrane, model DK2540F, is characterized by a mean pore size value of 0.5 nm. The active membrane area of the module is equal to 2.51m2 and the maximum allowable operating pressure is equal to 32 bar. Acting on the regulation valves V21 and V28 it is possible to set the desired operating pressure PEXT over the membrane maintaining the feed flow rate constant with a precision of 0.5 bar.

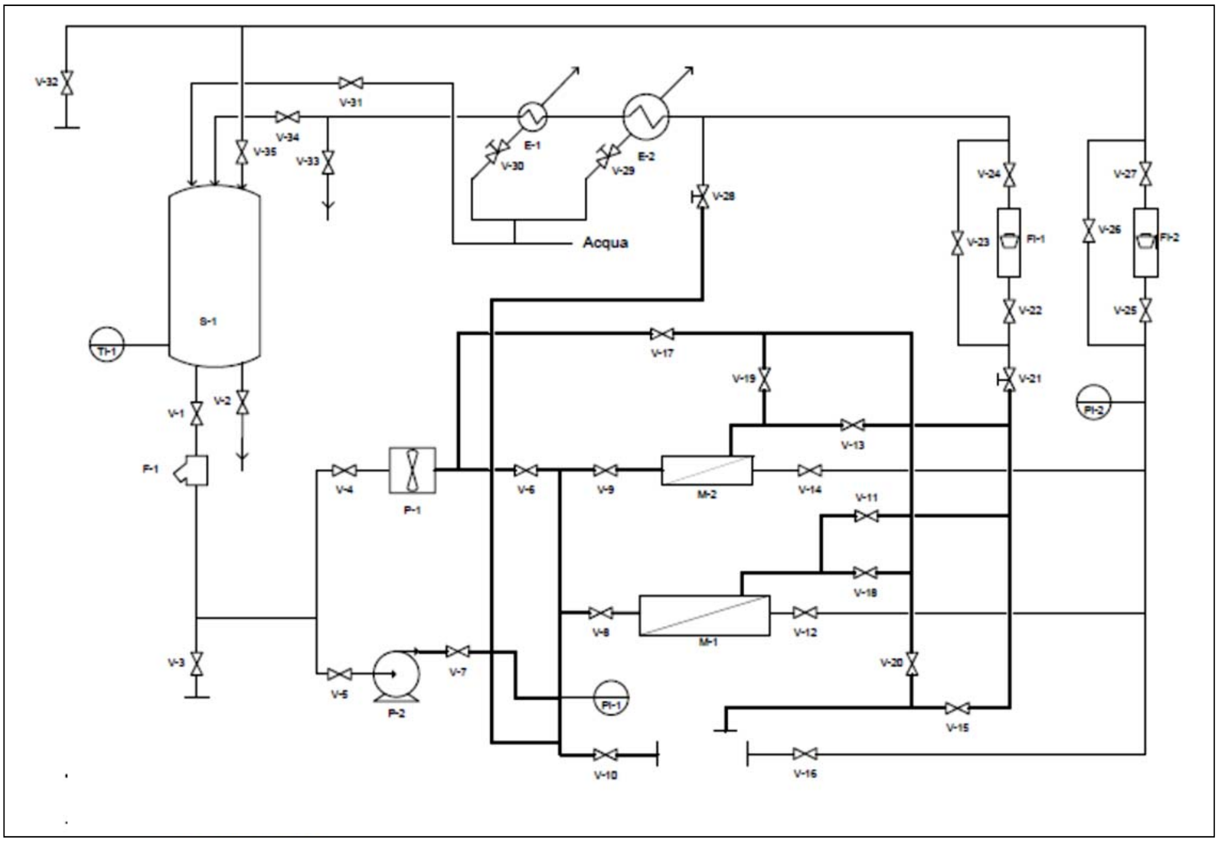

Figure 1 - Scheme and photograph of the pilot plant

Both permeate and concentrate streams are cooled down to the feedstock temperature, mixed together and recycled back to the feedstock. In this way, the feedstock composition is kept constant during the experimental campaign. The temperature was controlled in all experiments at the value of 20 ◦C±1 ◦C.

After each experiment the membrane was rinsed with tap water at least 30 min. If not necessary, the membrane module was stored directly in the membrane housing filled with fresh tap water, else put in a fresh tap water filled external storage tank. Sometimes chemical cleaning was additionally performed to rinsing by using a 1M NaOH solution in closed loop for 30 min.

2.3 Measurements

Critical flux can be measured by different procedures. In this work, all the critical flux determinations were carried out with the pressure cycling method proposed by Espinasse et al. (2002). The same Basically, the method consists of cycling the applied pressure up and down, and to check for the reproducibility of the permeate flux at same pressure values before and after the pressure changes. The highest pressure value at which the same permeate flux is obtained before and after a pressure cycle is the critical flux. After each measurement, the critical flux value JC and the correspondent TMP value were noted.

Moreover, COD was measured by means of a photometer LASA 100 combined to the COD Cuvettes LCK014 supplied by Dr. Hach Lange and electroconductivity EC was measured by the portable instrument 8706R1 supplied by Delta Ohm. Particle size measurements were performed by dynamic light scattering instrument Plus90 supplied by Brookhavn.

3. Results and Discussion

The adopted optimization method, previously developed by Stoller and Chianese, is described in detail elsewhere (2006). Briefly the method relays on the determination of the critical flux as function of two key parameters:

I. Chemical oxygen demand ("COD", expressed in mg/l), which is an indirect measurement of the organic matter concentration in the feedstock. This measurement is quantitative. An increase of this value has as consequence a reduction of the critical flux values of the membrane, by means of a logarithmic profile [14].

II. Particle size distribution ("PSD", expressed as #/l), measured by a dynamic light scattering device, which characterize the suspended matter and thus in case of this kind of wastewater the organic matter sizes in the feedstock. This measurement is qualitative.

Particles of certain size, compared to the average pore size of the membrane dp, strongly affects critical flux values (Stoller et al., 2007). An increase of the number of these particles interfering with the membrane leads to critical flux reductions. As a rule of thumb, particles with size between (1/10) dp and 10 dp may be considered interfering. In fact, PSD measurements allows to measure the percentage of the total suspended particles in the feedstock which may strongly interfere with the membrane's performances without giving any indication about the total suspended matter concentration. In combination with COD measurements, it is possible to evaluate the effective amount of suspended organics which interferes with the membrane.

By a simulation model it is possible to determine the optimized value of the permeate flux to adopt throughout the batch.

Nevertheless, the entire approach is based on the critical flux determination of the examined membrane system and the correlation of the critical flux value as function of the chosen key parameters. Critical flux can be measured by different procedures such as the pressure cycling method proposed by Espinasse et al. (2002).
A main problem during the determination of the critical flux was that the system did not exhibit a pure critical flux. In fact, below critical flux no fouling must be observed, as in the case of treating olive wash wastewater (see Figure 2). Here critical flux appears due to the slightly polluted nature of the stream, and therefore the weak interference of solutes with the membrane pores. But in the examined system, even at very low transmembrane pressure values (TMP), a long-term fouling was always observed and a permeate gap exists from the beginning (see Figure 3).

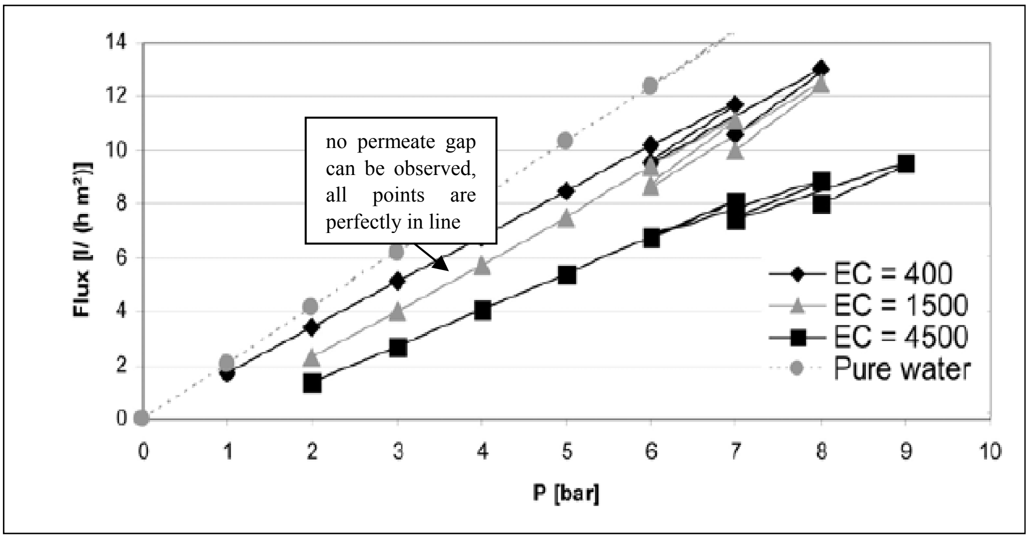

Figure 2 - Example of critical flux measurement on olive washing wastewater

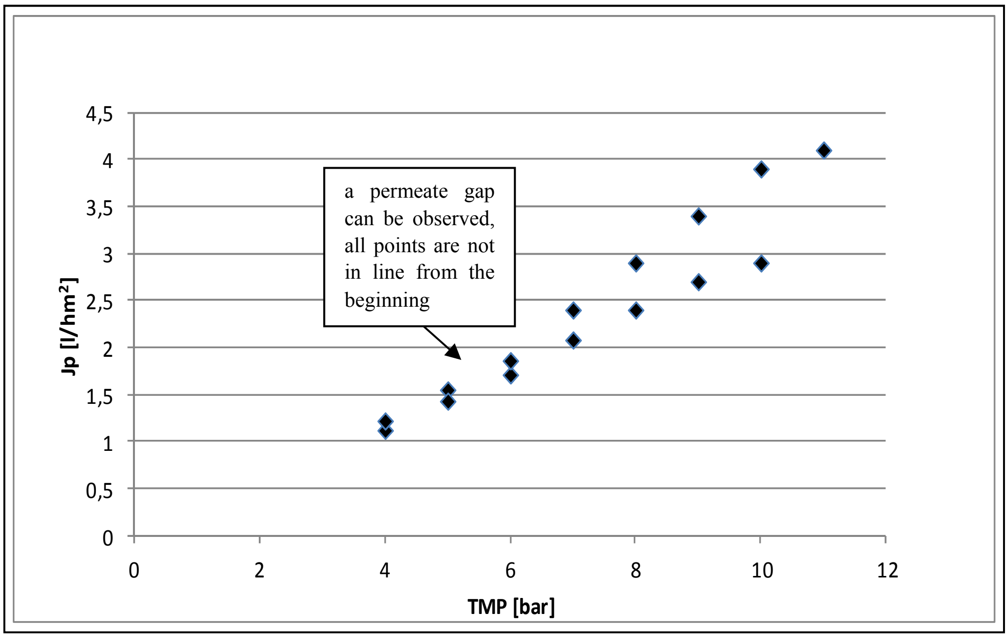

Figure 3 - Example of threshold flux measurement on OMWW

At that time, the threshold flux concept did not exist. The only available tool was the critical flux, and even it does not fit perfectly to observations, the authors decided to adopt the strategy to define the critical point the one that did give rise to a permeate flux gap lower than 5% when measured at the same TMP value before and after one pressure cycle.

Nowadays, this approach is known to be imprecise; nevertheless, by the same approach is was possible to use the membrane module for more than 3 years, suffering a pure water permeability loss of only 6.4% (Stoller, 2011). Therefore, the suggested approach by the Authors in the past appears to be somehow valid, a rule of thumb to use on wastewater membrane systems.

Concerning the critical flux Jc, hereafter used in terms of critical flux for irreversibility, the following fitting equations apply [8]:

$dm/dt = 0; Jp(t) \leq Jc$ (1)
$dm/dt = B\,(Jp(t) - Jc); Jp(t) > Jc$ (2)

where m is the permeability of the membrane, B is a fitting parameter and Jp(t) the permeate flux at time t. The corresponding transmembrane pressure value at Jc will be labeled hereafter as TMPc.

Concerning the threshold flux Jth, the proposed equations by Field et al. are as follows [15]:

$dm/dt = a; Jp(t) \leq Jth$ (3)
$dm/dt = a + b\,(Jp(t) - Jth); Jp(t) > Jth$ (4)

The corresponding transmembrane pressure value at Jth will be labeled hereafter as TMPth.

Eq.(3) can be integrated between a time point t_1 and t_2, and the following linear equation can be derived:

$m(t_2) - m(t_1) = \Delta m = a\,(t_2 - t_1)$ (5)

Permeate flux and permeability values are strictly connected by the following general equation:

$m(t) = Jp(t) / TMP(t)$ (6)

Merging together eq.(5) and eq.(6), the following relationship is obtained:

$$Jp(TMP,t_1) - Jp(TMP,t_2) = -\Delta Jp^* = a\ TMP\ (t_1 - t_2) \qquad (7)$$

valid in case the same TMP value is used at t_1 and t_2. It is possible to use different TMP values between t_1 and t_2 without invalidating eq.7: as long as the adopted TMP values remain below the threshold one, no effect on changes of the permeability loss rate should be observed. $-\Delta Jp^*$ is the expected permeate reduction if eq.3 holds, that is at subthreshold flux regimes, and must be compared to measured one equal to $-\Delta Jp$.

One important aspect to consider at this point is that the threshold flux determination requires the measurements of the permeate fluxes and the knowledge of the measurement time. Different measurement times will lead to different results. At the beginning, critical flux values were measured waiting for the complete development of membrane polarization and thus stabilization of the permeate flux. This measurement method was characterized by different measurement times of the pressure cycles, and since the Authors were not aware of the importance of this parameter, it was not noted during experiments: in this case, the recovery of the data in terms of threshold flux is not possible. Luckily, the method changed since year 2008, after years of continuous observations, where a new measurement strategy was adopted, that is to perform the permeate flux measurements after a fixed time sufficient to guarantee the stabilization of the permeate fluxes, equal to 30 minutes. In this case the measurement time is known and a recovery of the data in terms of threshold flux is possible.

At this point, the Author wanted to introduce the concept of boundary flux, which merge eq.(1) to eq.(4) into a single set. The introduction of the new boundary flux concept does not extend by addition of new theory or knowledge the critical and threshold flux concepts. On the other hand, it tries to simplify the use of these concepts in future works. Referring to one single concept will reduce sensibly the incorrect use of both the critical and threshold flux concepts, and this may be of help for a better communication among both academics and operators in membrane technologies.

It is interesting to notice that the threshold flux equations are similar to the critical flux equations and differ only by the presence of the “a” parameter. In fact, if the case of a = 0 is admitted, eq.(3) and eq.(4) may reduce to eq.(1) and eq.(2), respectively.

The parameter “a” value measures below threshold flux conditions the constant permeability loss rate of the membrane in time. If this value is equal to zero, no permeability will be lost in time and therefore no fouling is triggered. This is valid only below critical flux conditions, and therefore eq.(3) include eq.(1) if $a = [0,\infty)$.

Above critical and threshold flux conditions, fouling behaves in similar way by exponential permeability loss rates in time. Again, if $a = [0,\infty)$, eq.(4) fits eq.(2). The only difference between these systems is that in critical flux characterized systems fouling is not affected by the continuous presence of a constant fouling permeability loss rate as in threshold flux characterized systems. Beside this theoretical difference of the two systems, the Authors want to point out that this aspect is of limited practical importance, since the exponential part of eq.(2) and eq.(4) will quickly overwhelm the linear contribution of the parameter “a” in eq.(4).

Summarizing, both critical and threshold fluxes divide the operation of membranes in two regions: a lower one, where no or a small, constant amount of fouling triggers, and a higher one, where fouling builds up very quickly. By introducing a new flux, that is the boundary flux Jb, the previous equations may be written as:

$$dm/dt = -\alpha;\ Jp(t) \le Jb \qquad (8)$$
$$dm/dt = -\alpha + \beta\ (Jp(t) - Jth);\ Jp(t) > Jb \qquad (9)$$

where:

- α, expressed in [$L\ h^{-2}\ m^{-2}\ bar^{-1}$], represents the constant permeability reduction rate suffered by the system and will be hereafter called the sub-boundary fouling rate.
- β, expressed in [$h^{-1}\ m^{-2}\ bar^{-1}$], represents the fouling behavior in the exponential fouling regime of the system, and will be hereafter called super-boundary fouling rate.
- The corresponding transmembrane pressure value at Jb will be labeled hereafter as the boundary transmembane pressure, TMPb.

The method to measure the boundary flux is similar to the ones used to measure critical flux values, but needs a different approach in order to determine the value of α at first, the value of β successively (see Figure 4). The boundary point is the one t the lowest TMP value where the fitting by eq.(8) is lost.

Beside experimental data the extended method requires the use of eq.(8) and eq.(9) to separate the two operating regimes. Eq.(7) can be rewritten in terms of boundary flux as:

$$Jp(TMP,t_1) - Jp(TMP,t_2) = -\Delta Jp^* = \alpha\ TMP\ (t_1 - t_2) \quad (10)$$

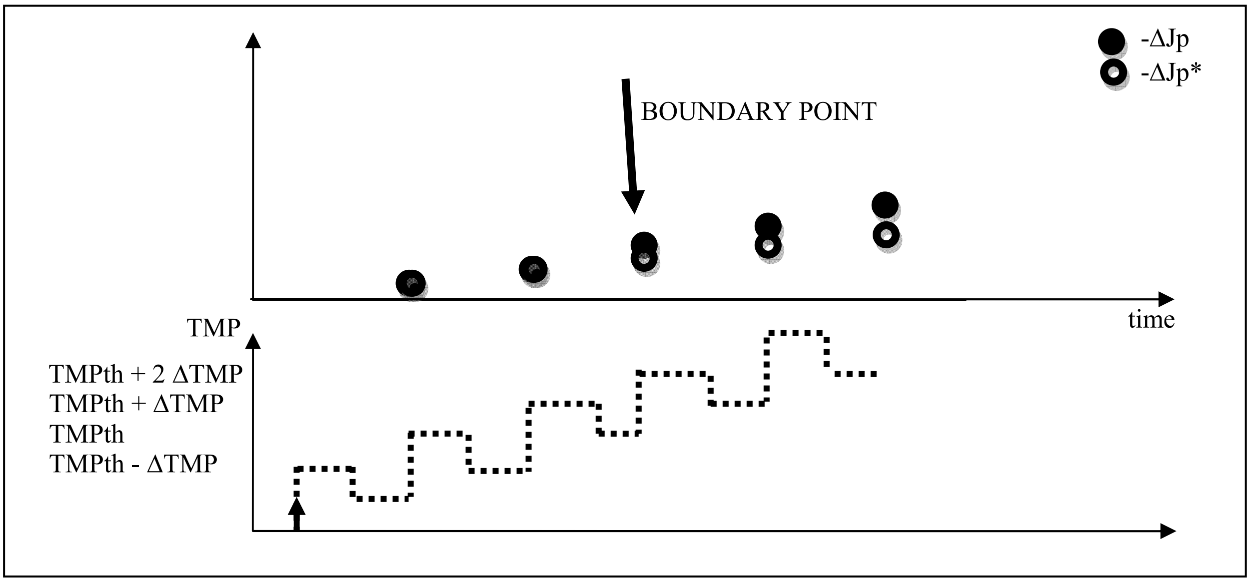

Figure 4 - Boundary point determination.

By introducing the boundary flux, no more distinction among critical and threshold flux must be performed, being the critical flux a particular case of the threshold flux and thus completely incorporated in the boundary flux concept.

The boundary fluxes were measured on the system from year 2008.
In Table 1, the critical flux measurements performed at June 2008 (A), July 2008 (B) and February 2009 (C) are reported in terms of the permeate flux before (Jp1) and after (Jp2) a pressure cycle. It is possible to use eq.(8) for the first data point to estimate the value of the sub-boundary fouling rate and eq.(10) to calculate -ΔJp*, that is the difference between this latter value and the one measured ΔJp (Table 2). In the first case, the critical flux was determined as soon as ΔJp% is greater than 5%; in the second case, the boundary flux is determined as soon as ΔJp-ΔJp* becomes positive.

Table 1 - Critical flux measurements performed at June 2008 (A), July 2008 (B) and February 2009 (C); critical point in bold

TMP [bar]	A			B			C		
	Jp1	Jp2	ΔJp%	Jp1	Jp2	ΔJp%	Jp1	Jp2	ΔJp%
5	9.32	9.30	0.2	9.56	9.54	0.2	8.54	8.52	0.2
6	11.19	11.16	0.3	11.47	11.43	0.4	10.29	10.25	0.4
7	13.05	12.98	0.6	**13.39**	**13.33**	**0.5**	**12.00**	**11.92**	**0.6**
8	**14.92**	**14.82**	**0.7**	15.30	13.69	10.5	13.71	12.20	11.1
9	16.79	14.94	11.0	17.21	15.32	10.9	15.43	13.65	11.6

Table 2 - Estimation of the value of the sub-boundary fouling rate and calculation of -ΔJp at June 2008 (A), July 2008 (B) and February 2009 (C); boundary point in bold*

	ΔJp - ΔJp*		
TMP [bar]	A α = 0.008	B α = 0.008	C α = 0.008
5	0.000	0.000	0.000
6	-0.018	-0.008	-0.008
7	-0.014	**-0.024**	**-0.004**
8	**-0,028**	+1.482	+1.382
9	+1.670	+1.710	+1.600

It is possible to observe that both analyses lead in all cases to the same value of TMP for critical and boundary flux, respectively. Therefore the wrong application of the critical flux by adopting the suggested approach by the authors gave in fact comparable results to the boundary ones.

This result can be justified by the different fouling behavior the membrane system exhibits in sub-boundary (eq.8) and super-boundary (eq.9) regimes: the permeate flux gap of 5% is quickly reached as soon as eq.9 becomes valid. Therefore, the definition of a safety zone of 5% was sufficient to separate the low fouling operating conditions from the high fouling one, and as a consequence, to identify the boundary flux as a threshold flux before it was introduced as theory.

4. Conclusions

The boundary flux concept is an interesting advance in membrane knowledge, shares all the characteristics of the critical and threshold flux theory, and can be used successfully during membrane plant design. Moreover, the merged concept helps to avoid the confusion among membrane technology operators to make a distinction between critical and threshold fluxes, which is not longer required.

The pressure cycling method appears to be suitable for boundary flux determination. Most important is the correspondent TMP value (TMPb), which appears to be more raible if compared to the boundary flux Jb changing as a function of time. Operating at or below boundary flux conditions results in worst case in the build-up of (mostly) reversible fouling, which can be periodically washed.

Although critical fluxes were measured, the addition of a constraint on the permeate flux gap has permitted to determine successfully the boundary flux, and as a consequence, to operate a nanofiltration membrane for three years, suffering only a performance loss of 6.4%.

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