International Journal of Environmental Engineering– IJEE
Volume 2 : Issue 1 [ISSN : 2374-1724]
Publication Date: 30 April, 2015

Comparison between two Moving Bed-Membrane BioReactors (MB-MBRs) for the treatment of real saline oily wastewater

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***Abstract*— The aim of the study is to evaluate the simultaneous effect of salinity and hydrocarbons on the performances of two parallel Moving Bed-Membrane BioReactor (MB-MBR) systems fed with real saline oily wastewater (slop). Different dilution factors were considered during the experiments. The first analysed MB-MBR system was characterized by the use of Linpor® carriers (MB-MBR_I), the other one by AnoxKaldnes™K1 carriers (MB-MBR_{II}); in both systems an ultrafiltration membrane was used. The results showed a decrease of biological removal efficiencies for both systems mainly due to the total petroleum hydrocarbons (TPHs) playing an inhibitory role in the biomass growth; this was confirmed by negligible efficiencies in the removal of the TPHs. The analysis of the extracellular polymeric substances (EPSs) has highlighted a reduction of bound EPSs and a simultaneous increase of the protein fraction of the soluble microbial products (SMPs) likely due to the cellular lysis, as response to the increase of salinity and hydrocarbons concentration. No biofilm formation has occurred in MB-MBR_{II} system while the MB-MBR_I system showed an opposite behaviour. This fact has affected the fouling deposition on the membrane surface, indeed a biofilm detachment was observed in the MB-MBR_I system which conditioned the irreversible cake deposition (showing an increase of 50% in the total resistance to filtration). On the other hand, the pore blocking was more evident in the MB-MBR_{II} system (up to 61% of total resistance to filtration), likely due to the occlusion of the membrane's porosity by the SMPs released into the mixed liquor through the cellular lysis.**

Keywords*— biofilm; EPS; fouling; MBR; salinity; slops; TPH

I. Introduction

Nowadays, the washing of oil tankers with sea water involves a high environmental impact. This activity produces mixtures of residual fuel oils and saline water, also called slops, in amounts of millions of tons per year. The slops, which are often transported in barges, contain several pollutants such as oils, hydrocarbons, surfactants and others hazardous substances for the environment. In this context, the International Maritime Organization (IMO) plays a relevant role regulating and preventing marine pollution by means of International Convention for the Prevention of Pollution from Ships, 1973, as modified by the Protocol of 1978 relating thereto (MARPOL 73/78) [1]. This regulation has defined the Mediterranean Sea as "special area", preventing the direct discharge of oils to the sea and forcing harbour authorities to implement wastewater treatments. Biological treatment of slops is an alternative method to physicochemical treatment but it must take into account the coexistence of salinity and high concentration of total petroleum hydrocarbons (TPHs). The salinity can inhibit the metabolism of microorganisms in activated sludge systems because of plasmolysis [2,3,4], but in highly saline environment, halophile microorganisms having considerable ability to purify saline wastewater can be selected [5]. The biological removal of TPHs is uncertain due to their toxicity towards bacteria [6], therefore biomass acclimation is required for any biological treatment [7]. An increasing interest in the use of membrane bioreactor (MBR) technology has been expressed in the last decades especially for the treatment of saline wastewaters [2,3,4,8] and for high salinity wastewaters contaminated by hydrocarbons [6,7,9]. The MBR system has many advantages acknowledged in literature including, among all, high effluent quality and the possibility to treat slowly biodegradable substances [10]. By adding suspended carriers in the mixed liquor of a conventional activated sludge reactor, a hybrid moving bed biofilm reactor (HMBBR) can be obtained having the coexistence of suspended and attached microorganisms [11]. This allows to achieve a higher biomass concentrations with the possibility to biodegrade the most recalcitrant compounds such those contained in slops. Coupling a MBR system with a HMBBR system or simply MBBR, it is possible to create new layouts respectively called MB-MBR and BF-MBR [11,12,13] which combine the benefits of both the technologies. Like all systems based on membrane technology, the above mentioned schemes present fouling problems whose minimization represented a great challenge for several years [14,15]. The aim of the present study is to evaluate the effect of simultaneous presence of salinity and hydrocarbons on the performances, both in terms of removal efficiencies and membrane fouling, of two parallel MB-MBR systems which differ for types of utilized carriers.

II. Methods

A. *Bench scale plants description*

The bench scale plants were built at the Laboratory of Sanitary and Environmental Engineering of Enna University (Italy); the plants were built adopting the same configuration of Di Bella et al.,[16] (Fig.1). In particular, the two plants

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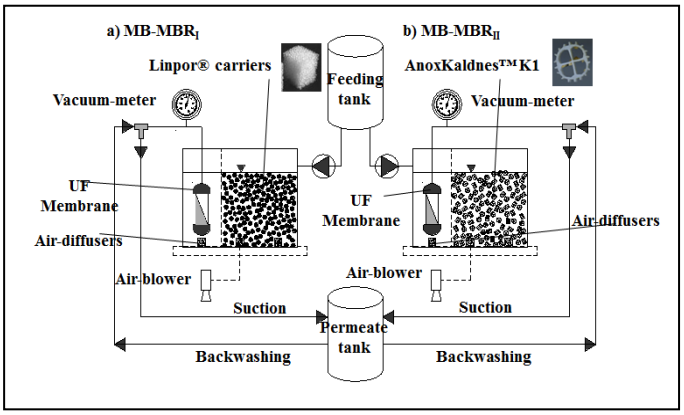

Figure 1. Layout scheme of bench scale plants: a) MB-MBR$_I$ with Linpor® carriers and b) MB-MBR$_{II}$ with AnoxKaldnes™K1 carriers.

were progressively adapted to the contaminants and salinity starting with highly diluted slops and progressively reducing the dilution factor. Both systems were characterized by a bioreactor of 14 L and by an ultrafiltration (UF) hollow fibre membrane module (Zee-Weed™01) having specific area equal to 0.093 m^2 and nominal porosity of 0.04 µm. The permeate flux was kept at about 15 $L \cdot m^{-2} \cdot h^{-1}$ and the hydraulic retention time (HRT) was equal to about 18 h. The membranes were periodically backwashed (every 5 min for a period of 1 min) by pumping a fraction of permeate back through the membranes module. The membranes were kept in a separated compartment from the rest of the reactor; a perforated wall separates the two compartments in order to avoid collisions of suspended carriers with UF modules. In the first reactor (indicated by MB-MBR$_I$) the mobile carriers were Linpor®: polyurethane cubic sponges with a 14 mm side and a 1000 m^2/m^3 specific effective carrier surface area. In the second reactor (indicated by MB-MBR$_{II}$), AnoxKaldnes™K1 were used: polyethylene cylindrical carriers with a 500 m^2/m^3 specific effective carrier surface area. The filling ratio was 31% for both systems. The bench scale plants have been operated for a period of about 60 days. The experimental campaign was divided in three different phases, each characterized by a different slop dilution factor to allow microorganisms acclimation (Phase I: no slop addiction, Phase II: 5% slop volume, Phase III: 10% slop volume). Starting from the Phase II, slop was mixed with synthetic solution containing sodium acetate, ammonium chloride and potassium diphosphate, in order to maintain a COD next to values of 1000÷1500 mg/l and in proportions to ensure minimum intake of C:N:P for bacterial metabolism in the respectively ratio of 100:5:1. The slops were sampled from barges of an oil coastal deposit in the Augusta harbour (Sicily) and subjected to a preliminary treatment of de-oiling, prior to biological treatment, in order to remove a large amount of non-biodegradable oils and greases. In Table I and Table II, the average main slop characteristics and the average operational parameters are respectively summarized. The MB-MBRs plants were inoculated with a mixed liquor suspended solids (MLSS) concentration almost equal to 3.3 $g \cdot L^{-1}$. In order to maintain constant the concentration of suspended solids within the systems between 7-8 $gMLSS \cdot L^{-1}$, occasionally sludge withdrawals were carried out from the systems.

TABLE I. WASTEWATER INLET CHARACTERIZATION

Parameter	Slop	Phase I	Phase II	Phase III
		0% Slop	*5% Slop*	*10% Slop*
COD ($mg \cdot L^{-1}$)	1566	1095 ± 346	1328 ± 200	1303 ± 205
TC ($mg \cdot L^{-1}$)	428	459 ± 84	521 ± 41	533 ± 29
IC ($mg \cdot L^{-1}$)	38	79 ± 31	113 ± 32	174 ± 56
TOC ($mg \cdot L^{-1}$)	390	380 ± 54	408 ± 73	359 ± 84
N-NH_4($mg \cdot L^{-1}$)	≈ 0	115 ± 37	167 ± 66	155 ± 75
Cl^- ($mg \cdot L^{-1}$)	23455	216 ± 5	1446 ± 121	2393 ± 260
Br^- ($mg \cdot L^{-1}$)	411	0	21 ± 5	41 ± 8
SO_4^{2-} ($mg \cdot L^{-1}$)	3541	0	143 ± 17	263 ± 48
TPH ($mg \cdot L^{-1}$)	30	0	1.5 ± 0.2	3.1 ± 0.5

TABLE II. OPERATIONAL PARAMETERS

Parameter	Phase I (0% Slop)		Phase II (5%Slop)		Phase III (10%Slop)	
	(I)	*(II)*	*(I)*	*(II)*	*(I)*	*(II)*
HRT (h)	17.5	18.7	17.7	17.3	17.9	17.5
Flux ($L \cdot m^{-2} \cdot h^{-1}$)	14.9	14.3	14.8	15.0	14.7	14.9
MLSS ($g \cdot L^{-1}$)	4.0	4.7	6.7	7.5	7.8	7.0
MLVSS ($g \cdot L^{-1}$)	3.2	3.7	5.6	6.3	6.3	6.1
SS Biofilm ($g \cdot L^{-1}$)	6.1	0	7.7	0	9.7	0
Operation time (d)	18		14		24	

B. *Analytical methods*

Throughout the period of operations, the influent wastewater, the mixed liquor and the effluent permeate have been sampled two times per week and analysed according to the Standard Methods [17]. In particular, the following parameters were measured: total and volatile suspended solids (MLSS and MLVSS), chemical oxygen demand (COD); ammonia nitrogen (NH_4-N), anions (nitrite (NO_2^-), nitrate (NO_3^-), chloride (Cl^-), bromide (Br^-), phosphate (PO_4^{3-}), sulphate (SO_4^{2-})) and total petroleum hydrocarbons (TPHs) concentration. The mixed liquor samples were firstly filtered through a 0.45 µm filter. The measures of all anions were carried out by means of ionic chromatography by ICS Dionex 1100. The total organic carbon (TOC) was measured by a TOC-V_{CSH} analyser that also provides the total carbon (TC) and the inorganic carbon (IC). The TPHs concentration was measured using a gas chromatograph equipped with a flame ionization detector (GC-FID, Agilent 6890N), after extraction of TPHs from samples with hexane. Periodically, samples of suspended carriers were taken and analised for total solids (TS) in order to evaluate the biofilm growth on carriers; for the details on the adopted procedure, the reader is referred to literature [18].

C. EPS analysis

The total Extracellular Polymeric Substances (EPS_T) are expressed as sum of bound EPSs and Soluble Microbial Products (SMPs), which represent the soluble portion of EPS_T, according to the following equation:

$$EPS_T = \text{bound } EPS_P + \text{bound } EPS_C + SMP_P + SMP_C \quad (1)$$

where the subscript symbol "P" or "C" indicates the relative content of proteins or carbohydrates respectively in the bound EPSs and SMPs. The SMPs were obtained by centrifugation at 5000 rpm for 5 min while the bound EPSs were extracted by thermal extraction method [19]. Carbohydrates in the EPS_T were determined according to the phenol–sulphuric acid method with glucose as the standard [20]. Proteins were determined by the Folin method with bovine serum albumin as the standard [21].

D. Resistances analysis

The membrane fouling was analised by employing the resistance in series model which is funded on cake layer removal with ''physical cleaning''. According to this model, the total resistance to filtration is defined by [22]:

$$R_T = R_m + R_{PB} + R_{C,irr} + R_{C,rev} \quad (2)$$

where: R_m is the intrinsic resistance of membrane; R_{PB} is the irreversible resistance due to membrane pore blocking; $R_{C,irr}$ is the fouling resistance related to superficial cake deposition removable by physical cleanings only; $R_{C,rev}$ is the fouling resistance related to superficial removable by ordinary backwashing. The total resistance to filtration (R_T) can also be described by the Darcy's law:

$$R_T = TMP / (J \cdot \mu) \quad (3)$$

where TMP is the transmembrane pressure (Pa), μ the permeate viscosity (Pa·s), and J the permeation flux ($m \cdot s^{-1}$). So R_m in (2) was estimated by measuring the water flux and the TMP of ultrapure water with new membrane module, using (3). Regarding the physical cleaning, it was necessary first extract the membrane from the reactor and in a second time wash it with ultrapure water; thus the cake layer on the membrane surface was removed according to the ''manual water rinsing'' [23]. Finally, the specific resistances to filtration were evaluated according to the following equations:

$$R_{PB} = R_{T1} — R_m \quad (4)$$

$$R_{C,rev} = R_{T2} — R_{T1} \quad (5)$$

$$R_{C,irr} = R_T — R_{T2} \quad (6)$$

where R_{T1} and R_{T2} are the total resistances measured after physical cleaning, according to (3), in ultrapure water and into the bioreactor respectively.

III. Results and discussions

A. Bench scale plants performances

Table III shows the average removal efficiencies for both bench scale plants. Given the reduced increase of salinity and hydrocarbons up to a maximum of about 2.4 $g \cdot L^{-1}$ of chloride and 3 $mg \cdot L^{-1}$ of TPHs respectively, no significant worsening of biological removal of organic matter, expressed as COD, was observed in both systems. In detail, both plants showed similar performances during each phase, reaching COD biological removal efficiencies in the range between 71% - 77%. Nevertheless, the combined effect of salinity and hydrocarbons is mainly reflected on the biological removal efficiencies of TOC that are reduced from approximately 91% to 77% for the MB-MBR_I and from about 92% to 71% for the MB-MBR_{II}. This apparent disagreement between the trends of COD and TOC is probably due to the different analytical method adopted, and to the influence on the reliability of COD measurements in presence of salinity [24]. The biological-physical removal efficiency of the COD and TOC has reached high values on average above 90%, indicating the filtering effect exercised by the membrane towards the dissolved organic compounds. Literature studies show that the metabolic inhibition by salinity takes over by higher saline concentrations compared to those of the present study [8,25]. This fact suggests that the inhibitory effect on the biomass in Phases II and III is mainly exerted by TPHs and the decline of removal efficiency of TOC can be related to the inability of biomass to biodegrade hydrocarbons, given an inadequate biomass acclimation to TPHs [7]. The previous considerations are confirmed by a negligible removal efficiency of TPHs up to about 8% and 5% for MB-MBR_I plant and MB-MBR_{II} plant respectively. This slight difference is likely due to phenomena such as adsorption and entrapment of hydrocarbons within the porosity of the sponge carriers in MB-MBR_I plant. Despite the autotrophic biomass is delicate and susceptible to saline shocks, it has not undergone any metabolic inhibition. Indeed, no worsening in the removal efficiencies of ammonium occurred and values on average above 95% in both systems were achieved.

TABLE III. BENCH SCALE PLANTS PERFORMANCES (AVERAGE VALUES).

	MB-MBR_I			**MB-MBR_{II}**		
	Phases			***Phases***		
	I	***II***	***III***	***I***	***II***	***III***
	0%	***5%***	***10%***	***0%***	***5%***	***10%***
ηCOD_{bio} (%)	74±8	72±3	77±5	77±8	71±1	77±9
$\eta COD_{bio\text{-}fis}$ (%)	88±9	89±7	95±4	88±9	91±3	95±5
ηTOC_{bio} (%)	91±5	89±4	77±2	92±5	87±3	72±2
$\eta TOC_{bio\text{-}fis}$ (%)	98±2	95±1	94±2	98±1	97±2	94±3
$\eta N\text{-}NH_4$ (%)	97±1	98±1	96±3	98±1	98±1	97±1
ηTPH (%)	-	< 1	8±2	-	< 1	5±1

B. Extracellular polymeric substances composition

Fig.2 shows the specific values (Fig. 2a, b) of the bound EPSs and the SMPs, in carbohydrate and protein components, for both systems. Starting from the Phase II, in which there is a dilution ratio with a percentage by volume of slop of 5%, a slight decrease of the bound EPSs is observed but not a significant increase in the SMPs (Fig.2a, b). Phase III showed a further reduction of bound EPSs and a simultaneous increase in the protein fraction of the SMPs, more marked in MB-MBR_{II} system. This behaviour is likely due to cellular lysis resulting in release of organic cellular constituents in response to simultaneous increase of salinity [25,3,16,11] and hydrocarbons concentration.

C. Fouling analysis

Regarding the membrane fouling behaviour, the AnoxKaldnes™K1 carriers showed no significant biofilm formation during the whole period, as reported in Table 2. These kinds of carriers require a pre-acclimation of the biological film from dispersed microorganisms, not aggregated in flocks. Differently, biofilm attachment on Linpor® carriers was facilitated by entrapment of biomass within the porosity of the supports. This difference is mainly reflected in the fouling analysis of the two systems. Fig.3 shows the evolution of total resistance to filtration (R_T) (Fig.3a), the fouling rate (FR) (Fig.3b) and the specific resistances (Fig.3c, d) for both systems. For the first 18 days (Phase I), the FR is higher in MB-MBR_{II} because the biomass, being only suspended, entirely impacts on the membrane surface and causes a considerable increase of the total resistance to filtration. More specifically, the formation of an irreversible cake-layer occurs. From the Phase II and for most of the Phase III (up to 49th day) there is a slightly higher FR for the MB-MBR_I plant with respect to the MB-MBR_{II}, likely due to the partial biofilm detachment from Linpor® carriers that altered the permeability of the cake layer. Probably, the detachment is due both to gradual saline inhibition towards the biofilm [11] and to reciprocal impact of the carriers. In terms of specific resistances the partial biofilm detachment implies that the greatest percentage contributions to the R_T, up to 49th day, in MB-MBR_I plant are represented by $R_{C,rev}$ and $R_{C,irr}$, respectively equal to about 34% and 50 % of R_T, while R_{PB}

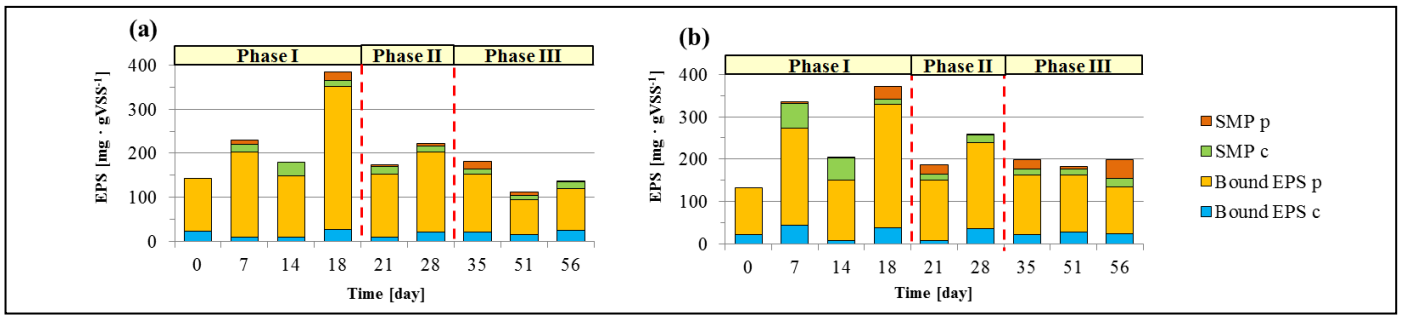

Figure 2. Specific EPSs distbution in MB-MBR_I (a) and in MB-MBR_{II} (b).

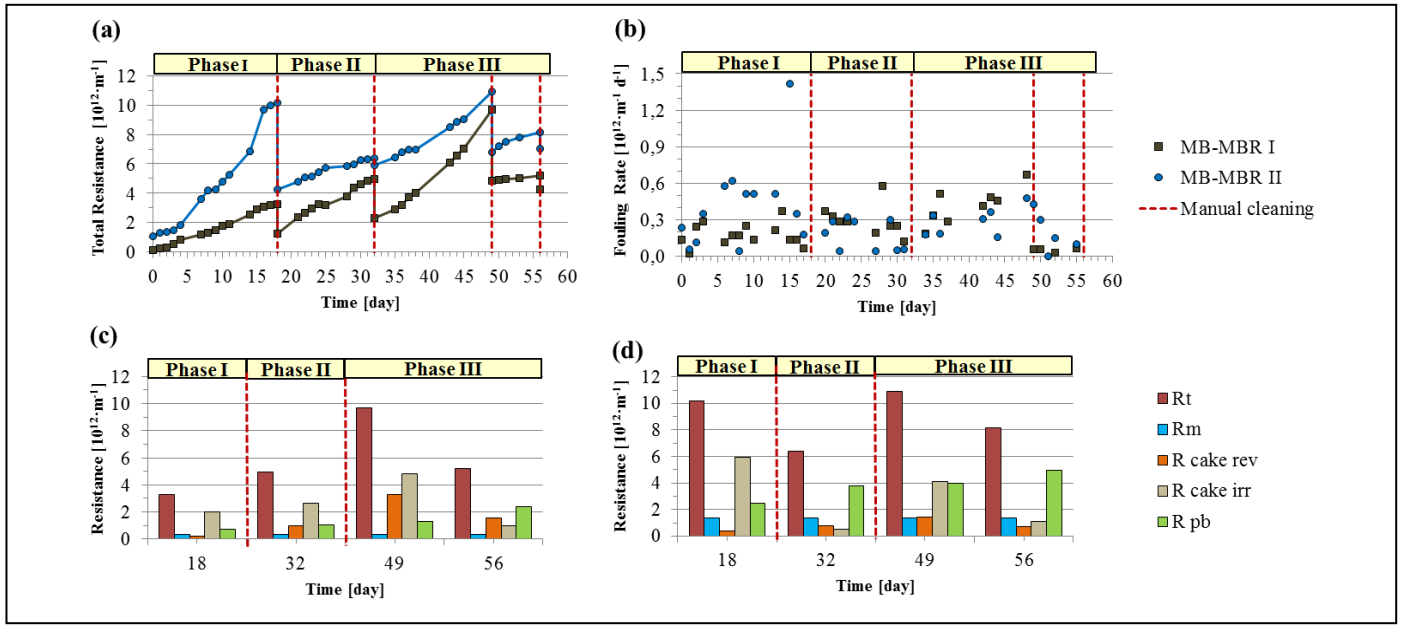

Figure 3. Total resistance fouling in the MB-MBR_I and in the MB-MBR_{II} (a); fouling rate in the MB-MBR_I and in the MB-MBR_{II} (b); specific fouling resistances in the MB-MBR_I (c) and in the MB-MBR_{II} (d).

is equal to approximately 13% of R_T (Fig.3c). From the 49th to the 56th day there is an increase in the contribution of R_{PB} (about 45% of R_T), due to the production of the SMPs, and a decrease of $R_{C,rev}$ and $R_{C,irr}$ linked to a lower biofilm detachment. In the MB-MBR_{II} plant, since the biofilm is negligible, the main contribution to the R_T is represented by R_{PB} (about 58% of R_T at day 32 and about 61% of R_T at day 56) due both to the increased production of the protein fraction of SMPs from cellular lysis , and to the lower deposition of reversible and irreversible cake.

IV. Conclusions

Two MB-MBR systems were investigated in order to evaluate the effect of simultaneous gradual increase of salinity and hydrocarbons concentration on the pollutants removal and membrane fouling. The two plants were characterised by different types of utilized carriers: Linpor® (MB-MBR_I) and AnoxKaldnes™K1(MB-MBR_{II}). Both plants provided good COD biological removal efficiencies, nevertheless a decrease of biological removal efficiencies measured as TOC was highlighted. This behaviour is likely due to the inhibitory effect on the biomass exerted by TPHs which require a proper acclimation to be removed. The increasing salinity and hydrocarbons concentration caused a release of SMPs, especially as protein fraction, due to cellular lysis as a response of microorganisms to the recalcitrant substrate. A biofilm formation and a partial detachment was observed in the MB-MBR_I system with subsequent deposition of irreversible cake. In the MB-MBR_{II}, since biofilm formation was negligible, the pore blocking was more pronounced. This study requires to be further investigated in the future researches treating an increasing percentage by volume of slop.

Acknowledgment

This research was funded by the National Operational Programme for Research and Competitiveness 2007–2013. Project "SIBSAC – An integrated system for sediments remediation and high salinity marine wastewaters treatment''.

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