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Removal of anionic surfactants by nanofiltration

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ABSTRACT

This work addresses the assessment of nanofiltration (NF) in terms of membrane characteristics, operating transmembrane pressure and feed composition for the maximal removal of anionic surfactants in wastewater from a detergent industry. Model solutions of linear alkylbenzene sulphonates (LAS) and sodium lauryl ether sulphates (SLES) covering a wide range of SLES/LAS ratios are used as surrogates of the wastewaters with 0.43 g l^{-1} of methylene blue active substances (MSAS). The NF experiments are carried out in a unit equipped with NF-90, NF-200 and NF-270 membranes (FilmTec Corp., USA). The applied pressure varied from 15 to 25 bar. The rejection coefficients to total organic carbon (TOC) are practically independent of pressure and are higher than 95% for all model solutions and higher than 92% for the wastewater. The SLES solutions have the highest permeation fluxes of 20–33, 121–207 and 242–371 kg h^{-1} m^{-2} for NF90, NF200 and NF270 membranes, respectively. The permeation fluxes for the other model solutions have intermediate values between the ones of the SLES solution and the ones of the wastewater. These present permeations fluxes as low as 10–11 kg h^{-1} m^{-2} for the NF 200 and the NF 270 membranes.

Keywords: Nanofiltration; Model solutions; Industrial wastewaters; Membrane; Adsorptive fouling; Anionic surfactants

1. Introduction

Surfactants are a class of industrially very important amphiphilic compounds. One of the characteristic properties of amphiphilic substances is that they tend to assemble at interfaces and therefore they are often referred to as surface-active agents. Another characteristic property of these substances is the formation of large aggregates (micelles). Surfactants are categorized into four groups depending on the charge on the head group: nonionic, anionic, cationic and amphoteric surfactants [1]. LAS, alkyl ethoxy sulphates (AES), SLES, alkyl sulphates (AS) and quaternary ammonium compounds (QAC) are the most commonly used commercial surfactants. LAS are the most extensively used for over 30 years [2].

The annual worldwide consumption of surfactants has been steadily increasing. The total amount of surfactants (without soaps) consumed in Western Europe in 2008 was 2.98 Mt, 1.413 Mt of which were non-ionic, 1.222 Mt were anionic, 0.254 Mt were cationic and 0.093 Mt were amphoteric, according to statistics published

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by the European Committee of Surfactants and their Organic Intermediates (CESIO) [3].

Surfactants have a negative impact on the environment during their life-cycle. Typically, production, formulation, the use phase, and the discharge phase are counted on their life-cycle [4].

In production plants such as the ones producing shampoo, liquid dishwasher detergent, shower gel and liquid soap, the plant equipments have to be cleaned and disinfected after every change of product for ensuring the hygiene and safety of products and for preventing contamination. The wastewaters generated from these cleaning operations are usually centrally collected and contain, besides surfactants, auxiliary substances, such as salts, dyes, perfumes, ethanol and others. These wastewaters may be treated by different processes, like biological treatment, foam fractionation, chemical precipitation, oxidation, adsorption and membrane processes [5–7]. However, the presence of surfactants in high concentration in biological stations produces foams that cause perturbations on the treatment process [6,8]. Furthermore, wastewaters containing a large amount of surfactants and high COD content cannot be easily treated either by conventional physico-chemical or biological processes [8,9].

After use, residual surfactants and their degradation products are also discharged to sewage treatment plants or directly to surface waters. LAS are aerobically well degraded, the formation of metabolites is well-known and display high levels of biodegradation in some waste water treatment processes [10]. However, LAS are believed not to degrade under anaerobic conditions and since much of the load into a sewage treatment facility is associated with suspended solids, they escape from aerobic treatment processes. In fact, LAS and other surfactants, as well as their degradation products, even when they are treated in a sewage treatment plant have been further detected in the environment. Surfactants enter the environment through the discharge of sewage effluents into surface waters and through the application of the sewage sludge on land as soil conditioner [4,11,12].

As a consequence of their widespread use, surfactants may persist in water systems at relatively high concentrations [11,13–15]. This poses severe environmental problems, as they are associated to interference with reaeration of water, which is low in dissolved oxygen. The search for efficient treatment processes remains still as a matter of research.

In detergent production plants, membrane processes are emerging as the leading contenders for processing water recycling and for the recovery of concentrated products from wastewater generated in the cleaning processes used in the batch production of detergents, or as a polishing step before the effluents are discharged [5]. In fact, membrane pressure-driven processes such as ultrafiltration (UF) and NF display adequate surfactant removals despite the membrane fouling problems. As expected, the UF surfactants removals [16–18] are lower than the NF ones [19,20]. Besides, for charged organic compounds, electrostatic attraction or repulsion forces between the component and the membrane influences the degree of fouling [21,22]. Van der Bruggen et al. [20] concluded that in surfactant removal by NF the anionic surfactants presented the least problems of membrane fouling. Arayici et al. [23] evaluated NF to the treatment of solutions containing high concentrations of anionic and nonionic surfactants and low dye and salt concentrations. The results showed no serious fouling of the NF membrane and a rejection efficiency of over 98% for anionic surfactants.

The present work evaluates in terms of membrane characteristics, operating transmembrane pressure and feed composition to the performance of the NF of an industrial wastewater. This wastewater is generated from the raw-material and product tanks from the washing and feeding equipment spillage and accidental product losses and the major components are two very common anionic surfactants: LAS and SLES. Model aqueous solutions of SLES and LAS and of its mixtures in the proportions of 1:5, 1:10, 1:20 are used as surrogates of the industrial effluent that has a total concentration of 0.43 g l^{-1} of MBAS. The MBAS parameter measures the blue colour intensity resulting from the association of the MBAS anion with the methylene blue cation. The surfactants discharge limits for the water body are fixed in 0.2 mg MBAS l^{-1} [24].

2. Experimental

2.1. Model solutions and industrial waste water

Commercial mixtures of surfactants consist of several tens to hundreds of homologues, oligomers, and isomers of anionic, nonionic, cationic and amphoteric compounds. It is for these reasons that the industry standard is to analyze for MBAS using LAS as the equivalent standard. Methylene blue, a cationic dye, will transfer into immiscible organic liquid upon equilibrium. This occurs through ion pair formation by the MBAS anion and methylene blue cation. The intensity of the resulting blue colour in the organic phase is a measure of MBAS. This method comprises three successive extractions from an acid aqueous medium containing excess methylene blue into chloroform ($CHCl_3$), followed by an aqueous backwash with two successive extractions of chloroform. The resultant blue colour in the $CHCl_3$ is measured by a spectrophotometry at 652 nm [25].

An industrial wastewater is collected directly from washing waters of a detergent industry based on LAS and SLES that account for a total concentration of 0.43 g l^{-1} of MBAS. Table 1 presents the characteristics of this industrial wastewater.

Binary aqueous model solutions of LAS and SLES were prepared from a LAS concentrate with 970 g l^{-1} MBAS and a SLES concentrate with 270 g l^{-1} MBAS, respectively. In order to act, in terms of MBAS, as surrogates of the industrial wastewater, the LAS and SLES solutions were prepared by dilution with deionised water with a final MBAS concentration of 0.4 g l^{-1}. Mixtures of SLES and LAS Solutions in the proportion of 1:5 (SLES/5LAS solution), 1:10 (SLES/10LAS solution) and 1:20 (SLES/20LAS solution) were prepared, all with a final total MBAS concentration of 0.4 g l^{-1}. The solutions were prepared with deionised water with a pH of 5.2 and a conductivity of 0.78 µS cm^{-1}. Table 2 presents the characteristics of all the model solutions.

2.2. Membranes

The three NF membranes, designated by NF90, NF200 and NF270, were supplied by Filmtec Corp (Minneapolis, MN, USA). The membrane characteristics according to the supplier are presented on Table 3.

They are in this work characterized in terms of the hydraulic permeability, L_p, and in terms of rejection coefficients to reference solutes – NaCl, Na_2SO_4 and glucose. The rejection coefficients to salts were determined in terms of conductivity and to glucose were determined in terms of TOC.

Table 1
Composition of the industrial wastewater

pH	9.8
Conductivity (mS cm^{-1})	15.2
COD (mgO_2 l^{-1})	8908
TOC (mgC l^{-1})	2344
MBAS (g l^{-1})	0.43
Total solids (g l^{-1})	14.55
Volatile solids (g l^{-1})	3.57
Total suspended solids (g l^{-1})	2.27
Volatile suspended solids (g l^{-1})	0.72

Table 3
Membrane characteristics [26]

Membrane type	NF90	NF200	NF270
	Polyamide thin-film composite		
Maximum operating temperature	45°C		
Maximum operating pressure	41 bar		
pH range, continuous operation	2–11	2–11	3–10
pH range, short-term cleaning (30 min)	1–12	1–12	1–13
Maximum feed silt density index	SDI 5		
Free chlorine tolerancec	<0.1 ppm		
Solute passage (%) $CaCl_2$	5–15	50–65	40–60
Solute passage (%) $MgSO_4$	<3	<3	<3

2.3. Nanofiltration permeation experiments

The NF experiments were performed in laboratory flat-cell units, previously described by Afonso and Pinho [27]. The membrane surface area of the permeation cells were 13 × 10^{-4} m^2. The NF setup is schematically presented in Fig. 1.

Membrane conditioning was carried out through the circulation of pure water (conductivity <1 µS cm^{-1}) pressurized at 30 bar for 2 h. This avoided pressure effects on the membrane structure in subsequent experiments. The NF experiments were carried out in total recirculation mode, where the permeate and the concentrate are recirculated to the feed tank. Permeation experiments of aqueous model solutions of anionic surfactants (LAS and SLES) and of an industrial wastewater were carried out in order to obtain the variation of permeate fluxes

Table 2
Characterization of the model solutions

	LAS	SLES	SLES/5LAS	SLES/10LAS	SLES/20LAS
pH	2.43	5.38	3.24	3.65	4.11
Conductivity (µS cm^{-1})	438	75.8	135.4	98.8	83.4
TOC (mg C l^{-1})	261.4	204	215.5	209.7	206.9
COD (mg O_2 l^{-1})	1007	791	834.2	812.6	801.8
MBAS (g l^{-1})	0.4	0.4	0.4	0.4	0.4

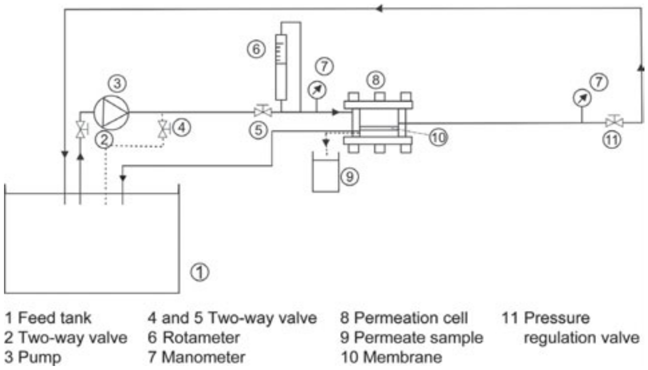

Fig. 1. Scheme of the NF installation [28].

and of the TOC rejection coefficients with the transmembrane pressure ranging from 15 to 25 bar. The rejection coefficients to a particular parameter (conductivity and TOC content) were determined by Eq. (1), where C_{feed} and $C_{permeate}$ are the conductivity or TOC content in the feed and in the permeate, respectively.

$$f\,(\%) = \left(C_{feed} - C_{permeate}\right)/C_{feed} \qquad (1)$$

The operating conditions were: temperature 25°C, transmembrane pressure ranging from 15 to 25 bar and feed flow rate of 90 l h^{-1}. The initial volume of feed solution for all experiments was 5 l. The membranes were rinsed with deionised water before and after the experiments.

2.4. Analytical methods

TOC analysis were performed in a TOC Analytical Aurora model 1030 analyzer and previously calibrated with potassium hydrogen-phtalate.

Conductivity and pH were determined by electrometry, chemical oxygen demand and MBAS by colorimetry and total solids, volatile solids, total suspended solids and volatile suspended solids by gravimetry. All these determinations were done accordingly to the Standard Methods for the Examination of Water and Wastewater [25].

3. Results and discussion

Fig. 2 displays the hydraulic permeability, L_p, of the NF membranes tested. Pure water permeation fluxes (PWP) were measured at transmembrane pressures (ΔP) of 10, 20 and 30 bar. The slope of the straight lines, PWP versus ΔP yields the L_p of 4.3, 8.3 and 13.6 kg h^{-1} m^{-2} bar^{-1} for NF90, NF200 and NF270, respectively.

The rejection coefficients to reference solutes – NaCl, Na_2SO_4 and glucose are presented in Fig. 3. For the glucose neutral solute, the rejection coefficients decrease with the increase of the membrane hydraulic permeabilities

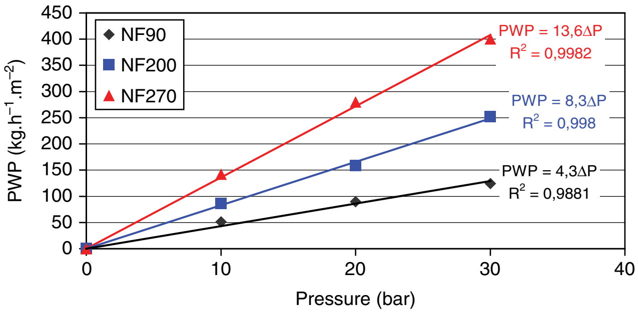

Fig. 2. Hydraulic permeability of NF90, NF200 and NF270 membranes.

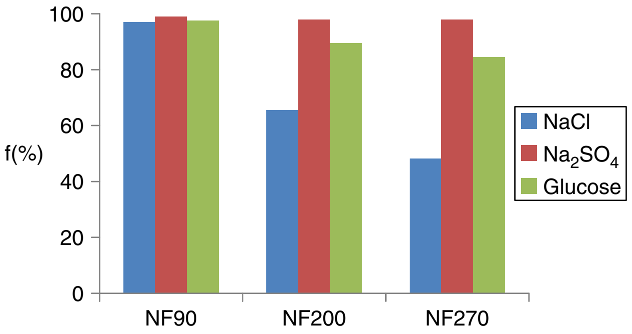

Fig. 3. Rejection coefficients to reference solutes – NaCl, Na_2SO_4 and glucose of NF90, NF200 and NF270 membranes. Transmembrane pressure = 15 bar and feed flow rate = 90 l h^{-1}.

due to the dominant role of the steric hindrance mechanisms. For the ionic species of the NaCl and Na_2SO_4 salts, the electrostatic mechanisms are the dominant ones. As the membranes are characterized by a negative surface charge distribution, this means a stronger interaction with the bivalent anions and therefore higher rejection coefficients to Na_2SO_4 [29,30].

The characterization of the industrial effluent in Table 1 shows a concentration of MBAS of 0.43 g l^{-1}. This MBAS concentration, which is an index of biodegradability, is mainly associated to LAS and SLES. The five model solutions described in the experimental section were prepared as a surrogate of this industrial wastewater in terms of biodegradability.

The apparent rejection coefficients to the global parameter of TOC are displayed in Table 4 for the industrial wastewater and for the Model Solutions.

For the tighter membrane, NF90, the rejection coefficients are 97 or 98% both for industrial effluent and the model solutions. For the NF200 and NF270 membranes and for the model solutions the rejections coefficients have the same values as for the NF90 membranes, whereas for the industrial wastewater the rejection coefficients are 93% and 94%. One can therefore conclude

Table 4
Apparent rejection coefficients, f_{TOC}, for the industrial wastewater and for the model solutions

	NF90				NF200				NF270			
	15 bar	17.5 bar	20 bar	25 bar	15 bar	17.5 bar	20 bar	25 bar	15 bar	17.5 bar	20 bar	25 bar
	f_{TOC} (%)				f_{TOC} (%)				f_{TOC} (%)			
Industrial wastewater	98	98	98	99	93	93	94	94	92	93	93	93
LAS solution	97	98	98	98	97	97	98	97	97	97	97	97
SLES solution	97	97	97	97	97	98	97	97	97	96	96	96
SLES/5LAS solution	96	97	98	98	96	96	97	97	96	97	97	97
SLES/10LAS solution	99	98	98	98	97	98	98	97	98	97	97	97
SLES/20LAS solution	98	97	97	97	97	98	98	98	98	98	97	97

that in the industrial wastewater there are other organic compounds, with lower molecular mass than the ones of LAS and SLES, contributing to the TOC. Although being lower than the values of 97–98% for the model solutions, they are higher than the rejection coefficients of the NF200 and NF270 membranes to glucose, meaning that these compounds have higher molecular mass than the one of glucose.

Figs. 4–6 display the permeation fluxes, J_p, versus the transmembrane pressure, ΔP, for the NF90, NF200 and NF270 membranes, respectively.

Except for the permeation of the industrial wastewater through membranes NF200 and NF270, the permeation fluxes, J_p increase always linearly with the transmembrane pressure, ΔP. The ratios of the slope describing this linear variation, J_p versus ΔP, by the slope of the PWP flux versus ΔP (hydraulic permeability) are shown in Table 5.

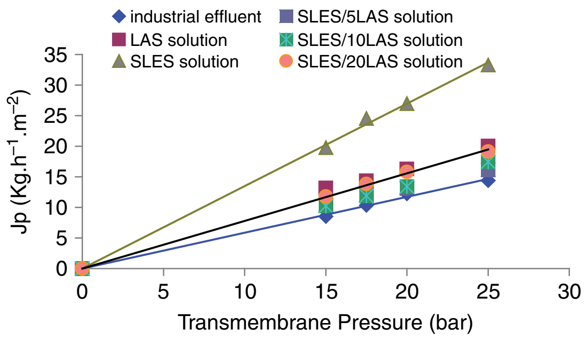

Fig. 4. Variation of permeate flux with transmembrane pressure—NF 90 membrane.

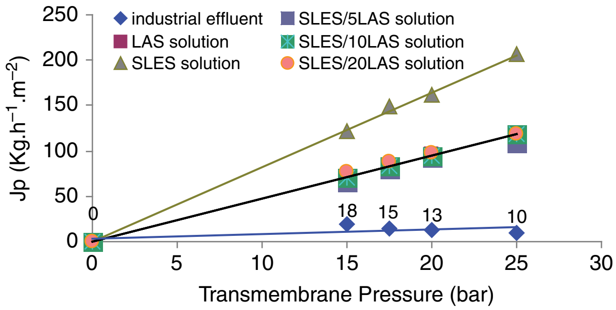

Fig. 5. Variation of permeate flux with transmembrane pressure—NF 200 membrane.

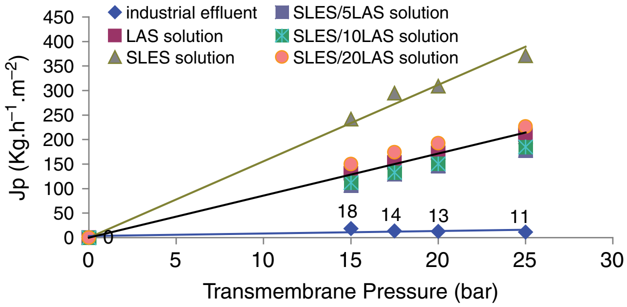

Fig. 6. Variation of permeate flux with transmembrane pressure—NF 270 membrane.

For all the membranes, the permeation fluxes for the SLES solutions are always higher than the ones for the other model solutions and for the industrial effluent. This is verified over all the pressure range. For the SLES solution, $J_p = 1.3\Delta P$ for the NF90, $J_p = 8.2\Delta P$ for the NF200 and $J_p = 13.6\Delta P$ for the NF270. For the membrane NF90 the slope of 1.3 is practically one third of the value of the corresponding hydraulic permeability, whereas for the membranes NF200 and NF270 the slopes for the SLES

Table 5
Permeation fluxes decline ratios (PFDR) for model solutions and industrial effluent. PFDR = slope of J_p versus ΔP/hydraulic permeability

Solutions	PFDRs		
	NF90	NF200	NF270
SLES solution	0.30	0.99	1.0
LAS solution	0.18	0.54	0.49
SLES/5LAS solution	0.18	0.54	0.49
SLES/10LAS solution	0.18	0.54	0.49
SLES/20LAS solution	0.18	0.54	0.49
Industrial effluent	0.13	–	–

permeation are practically equal to the ones of pure water permeation. For all membranes and over all the transmembrane pressure range, the permeation fluxes for the other model solutions, LAS solution, SLES/5LAS, SLES/10LAS and SLES/20LAS solutions have intermediate values between the ones of the SLES solution and the ones of the industrial effluent. In fact, the linearity of these fluxes with the transmembrane pressure is quantified by a single straight line with the same slope, independently of the fact of being a LAS pure solution or mixtures SLES/LAS.

The industrial effluent permeation patterns differ from the ones of the model solutions. The linear increase of permeation fluxes versus the transmembrane pressure is only observed for the NF90 membrane. This variation is characterized by a straight line with a slope of 0.58 kg h^{-1} m^{-2} bar^{-1}, whereas the SLES/LAS mixture's permeations have a slope of 0.78 kg h^{-1} m^{-2} bar^{-1}. For the other two membranes, NF200 and NF270 the permeation fluxes of the industrial effluent drop drastically and do not vary very much with the transmembrane pressures, and even display a slight decrease of the permeation fluxes with the increase of the transmembrane pressure. This may be attributed to the existence of components, besides LAS and SLES that cause severe fouling.

Table 1 shows that the conductivity of 15 mS cm^{-1} for the industrial effluent is 40–200 times higher than the conductivity of the model solutions (Table 2). The osmotic pressure, calculated in terms of NaCl equivalents to the conductivities of the model solutions, yielded values that are two or three orders of magnitude lower than the operating pressure values (15–25 bar) and therefore can be neglected. The osmotic pressure calculated in a similar way to the industrial effluent yields a value of approximately 7 bar. At a first analysis, one could consider that this high osmotic pressure could justify the drastic decrease of the industrial effluent permeation fluxes over all range of operating pressures. However, the membranes characterized by higher hydraulic permeabilities, NF200 and NF270, where the effect of osmotic pressure differences should be less pronounced, presented permeation fluxes for the industrial effluent one order of magnitude lower than the ones of the model solutions (Figs. 5 and 6). This is in contrast with the tighter membrane, NF90, where the permeation fluxes of the industrial effluent are of the same order of magnitude of the model solutions (Fig. 4). According to the NF experiments carried out by Drewes et al. [31] the effect of membrane fouling was found to be dependent upon the membrane evaluated.

The literature refers that the pH also plays an important role on the permeation flux behaviour. According to Kaya et al. [5], the flux decline and fouling of a membrane are significantly affected by the pH of the wastewater generated on detergent industry. Van der Bruggen et al. [20] also observed differences in NF membrane fouling according to the solution pH. The model solutions studied here have an acidic pH, while the industrial effluent presented a pH equal to 9.8. This difference could also explain the lower fluxes of the industrial effluent.

However, despite the potential effect of osmotic pressure and pH on flux decline, there is a clear evidence of fouling agents adsorbed to the membranes with higher hydraulic permeabilities, NF200 and NF270. This fouling acts as an additional resistance, making the permeation fluxes to decrease with the increase of pressure. The absence of this fouling phenomenon on the NF90 membrane that besides combines higher rejection coefficients makes of this membrane the best choice for the processing of this industrial effluent.

4. Conclusions

The NF for the removal of anionic surfactants from an industrial wastewater with a MBAS content of 0.43 g l^{-1} is assessed with the NF90, NF200 and NF270 membranes from FilmTec Corp (USA) and with transmembrane pressures (ΔP) ranging from 15 to 25 bar. Model solutions of LAS and SLES covering a wide range of SLES/LAS ratios are used as surrogates of the wastewaters in terms of the MBAS content.

The rejection coefficients to TOC are practically independent of pressure and are higher than 95% for all model solutions and higher than 92% for the wastewater.

The SLES solutions have the highest permeation fluxes of $J_p = 1.3\Delta P$, $J_p = 8.2\Delta P$ and $J_p = 13.6\Delta P$ for NF90, NF200 and NF270 membranes, respectively. The permeation fluxes for the SLES/5LAS, SLES/10LAS and SLES/20LAS solutions have intermediate values between the ones of the SLES solution and the ones of the wastewater. For the NF90 membrane the wastewater permeation fluxes increase linearly with ΔP whereas for the NF200 and NF270 they decrease with the pressure increase.

The NF90 membranes yield TOC removal rates of 98% and at operating pressures of 20 bar yield permeation rates comparable to the ones of the NF200 and NF270 membranes.

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