Microfiltration of vinasse: sustainable strategy to improve its nutritive potential
Míriam C. S. Amaral, Laura H. Andrade, Luzia S. F. Neta, Natalie C. Magalhães, Fábio S. Santos, Gabriel E. Mota and Roberto B. Carvalho

ABSTRACT
The purpose of this article was to evaluate and establish microfiltration (MF) operating conditions for vinasse (ethanol industries wastewater also known as stillage, slop, distillery effluent or dunder) concentration aiming to improve the use of its nutritive potential. The operating conditions influence permeate flux that has been evaluated by monitoring the flow rate profile during the operation on bench scale in different conditions (feed pH, aeration condition and recovery rate). From the results found, the process scale up was then effected. The bench scale findings showed that the vinasse microfiltration under air flow of 0.5 m³.h⁻¹ between membrane fibers, with no pH adjustment, and recovery rate of 93% produced two flows, one of permeate that may be used to wash the sugarcane during the ethanol production processing, and the other of concentrate that contains a high organic compounds and nutrients concentration. This concentrate has additional potentiality of being used as organic compound supplement in contaminated soil bioremediation, and as a supplier of microbial biomass or substrate for biosurfactant production.

Key words | fertigation critical flux, microfiltration, vinasse

INTRODUCTION
Brazil is the world’s number two ethanol producer and uses sugarcane as its feedstock. However, this high ethanol production has implications for the generation of high volumes of vinasse which represents significant disposal or treatment problems. According to Haandel (2005), for each 1 liter of ethanol, approximately 15 liters of vinasse are produced. Vinasse is characterized by extremely high chemical oxygen demand (COD) (22,000–100,000 mg.L⁻¹) and biochemical oxygen demand (BOD) (13,000–50,000 mg.L⁻¹), besides low pH, strong odor and dark brown color (España-Gamboa et al. 2011). In addition to high organic content, distillery wastewater contains nutrients such as nitrogen (1,660–4,200 mg.L⁻¹), phosphorus (225–3,038 mg.L⁻¹) and potassium (9,600–17,475 mg.L⁻¹) (Mahimairaja 2004).

Among the alternatives for vinasse disposal developed around the world, fertigation is the most commonly used. This practice has partially or even fully replaced the use of chemical fertilizers, particularly those containing phosphorus. However, the direct application of vinasse in the soil may cause salinization, leaching of metals present in the soil into groundwater, changes in soil quality due to nutrient content unbalance, particularly manganese (Agrawal & Pandey 1994), alkalinity reduction, crop losses (Kumar & Viswanathan 1991), increase of phytotoxicity and unpleasant odor (Santana & Fernandes Machado 2008). Then, some environmental parameters must be taken into account regarding fertigation, such as soil type, distance from it to water bodies, soil field capacity (water retention) and percentage of salts in the soil (Laime 2011). Studies conducted by Coopersucar, one of the cooperatives of sugarcane producers in Brazil, and Penatti et al. (1999) indicated that amounts up to 300 m³.ha⁻¹ of vinasse containing potassium levels between 3 and 4 kg.m⁻³ of vinasse, regardless of the type of soil, do not alter the soil properties physically, chemically or biologically.

The concentration of sugarcane vinasse is an alternative for the use of this residue, since fertigation cannot always dispose of the total volume of vinasse produced. The vinasse volume may be reduced by dehydration by using evaporator...
systems. According to Gomes et al. (2012), the substances contained in natural vinasse that account for its fertilizing effects will be present in the concentrated product. The concentrated product obtained in this process may be used as input to produce feedstuff, as fuel to generate energy or as a fertilizer. Improving vinasse fertilization quality by concentration may reduce the transportation costs with tanker trucks, and broaden the scope of vinasse use, particularly when the fertigation is unfeasible by piping systems (ANA 2009). High energy demand is probably the main constraint of vinasse concentration (Christofoletti et al. 2013). Furthermore the vinasse evaporations have problems associated with the fast incrustation of evaporators and spontaneous crystallization as the solids concentration increases (Rodrigues 2008).

Membrane filtration such as microfiltration (MF) has been used in many different sectors of industrial activity and is one method that could lead to improve value of vinasse. Thus it may offer an alternative to evaporation (Mahimairaja 2004; Santana & Fernandes Machado 2008; Gomes et al. 2012). Using membranes, the permeate stream from membrane filtration could be recycled to the process and concentrate could be used in fertigation or dried and fed to animals.

Microfiltration is a less energy-intensive process (7–9 kJ. kg\(^{-1}\) H\(_2\)O removed) compared with the triple-effect evaporation, that requires about 1,300 kJ. kg\(^{-1}\) H\(_2\)O removed (Rausch & Belyea 2006). MF membranes have also been shown to effectively remove suspended solids from vinasse (Arora et al. 2011). However, the operating conditions need to be optimized in order to facilitate the real scale application of these systems. In order to reduce capital and operational costs, pressure, temperature, permeate recovery rate and cleaning, among others parameters, must be adjusted to provide the best permeate flux.

Thus, the aim of this study was to evaluate the most effective operating conditions for the vinasse concentration by microfiltration. This study was performed on both bench and pilot scale.

**MATERIALS AND METHODS**

**Analytical methods**

Vinasse was characterized according to Standard Methods for the Examination of Water and Wastewater (APHA 2005) regarding the following physicochemical parameters: COD (5220B), solids (2540), total organic carbon (TOC) and total nitrogen (5310B – Shimadzu TOC-VCPH analyzer), color (2120B – Hach DR 3900 Spectrum Photometer), conductivity (Hanna conductivity meter HI 9835) and ions by ion chromatography ( Dionex ICS-1000 ion chromatography, equipped with column type IonPac AS22 and IonPac CS12A).

**Sampling and vinasse characterization**

Vinasse samples were obtained from a distillery located in the state of São Paulo, Brazil, which produces ethanol from fermentation of sugarcane juice. The samples were kept refrigerated at a temperature of 4 °C. The physicochemical composition of vinasse is shown in Table 1.

**Bench and pilot scale experimental setup**

Figure 1 shows the schematic drawing of the submerged bench and pilot scale MF system used. The system was composed of three tanks (feed storage tank, membrane tank and permeate storage tank with volumes of 13.4, 30 and 4 liters and 2,000, 400 and 200 liters for bench and pilot scale, respectively), a diaphragm pump was used to perform both microfiltration and backwash, three-way solenoid valves, level sensor, needle valves for flow adjustment, rotameters for permeate, backwash and air flows measure,

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Unit</th>
<th>Raw vinasse</th>
</tr>
</thead>
<tbody>
<tr>
<td>COD</td>
<td>mg.L(^{-1})</td>
<td>39,755</td>
</tr>
<tr>
<td>Color</td>
<td>Hazen</td>
<td>18,407</td>
</tr>
<tr>
<td>pH</td>
<td>–</td>
<td>4</td>
</tr>
<tr>
<td>Conductivity</td>
<td>μS.cm(^{-1})</td>
<td>3,733</td>
</tr>
<tr>
<td>Total solids</td>
<td>mg.L(^{-1})</td>
<td>13,678</td>
</tr>
<tr>
<td>Total volatile solids</td>
<td>mg.L(^{-1})</td>
<td>11,156</td>
</tr>
<tr>
<td>Total fixed solids</td>
<td>mg.L(^{-1})</td>
<td>2,522</td>
</tr>
<tr>
<td>Total suspended solids (volatile suspended solids)</td>
<td>g L(^{-1})</td>
<td>2.9 (2.8)</td>
</tr>
<tr>
<td>Volatile fatty acids</td>
<td>mgHAc.L(^{-1})</td>
<td>2,077</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>mg.L(^{-1})</td>
<td>81</td>
</tr>
<tr>
<td>Phosphate</td>
<td>mg.L(^{-1})</td>
<td>158</td>
</tr>
<tr>
<td>Sulfate</td>
<td>mg.L(^{-1})</td>
<td>763</td>
</tr>
<tr>
<td>Potassium</td>
<td>mg.L(^{-1})</td>
<td>1,665</td>
</tr>
<tr>
<td>Chloride</td>
<td>mg.L(^{-1})</td>
<td>394</td>
</tr>
<tr>
<td>Calcium</td>
<td>mg.L(^{-1})</td>
<td>315</td>
</tr>
</tbody>
</table>
pressure indicator, and a skid with an electric panel for the automatic control of permeation and backwash operations (Figure 1). It used a submerged hollow fiber module with a selective outer layer consisting of polyetherimide, with an average pore size of 0.4 μm, and total surface area of 0.045 and 3.5 m² for bench and pilot scale, respectively, supplied by PAM Membranas Seletivas Ltda (Rio de Janeiro, Brazil).

Evaluation of the operating conditions in bench scale

The effect of operating conditions on the microfiltration performance was assessed by monitoring permeate flow rate profile during the operation in each condition evaluated, which were feed pH, aeration condition, transmembrane pressures and recovery rate. The tests were carried out with the permeate and concentrate streams returning to the feed tank in order to maintain the same feeding condition (except for the recovery rate test, in which the concentrate stream returned to the feed tank). In the case of collecting permeate sample for analysis, an equivalent amount of the previously produced permeate was replaced to keep the feed specifications unchanged.

Since vinasse pH is low, effluent neutralization would be required for post biological treatment or wastewater reuse. The pH adjustment may be performed before or after the primary treatment, that is, microfiltration. Thus, permeate flux was monitored at 0.2 bar for the microfiltration of raw effluent (pH 4.76) and neutralized effluent (pH 7.0). The pH adjustment was performed by CaO addition. Permeate samples obtained with raw and neutralized vinasse were also characterized by COD, total solids and color.

Most of the submerged modules have an aeration constituted by air diffusers positioned at the bottom of the membrane tank. This conventional module aeration configuration was compared with another one, in which an aeration system was uniformly distributed among membrane fibers. In both configurations, airflow of 0.5 Nm³.h⁻¹ was applied. Permeate flux and permeate quality, measured by COD, total solids and color, were analyzed.

In order to find the most effective permeate recovery rate, 26 liters of effluent were supplied to the MF system. The permeation was performed along with a continuous permeate removal, and under pH and aeration conditions set during the preliminary tests and pressure of 0.2 bar. The flow rate and the total accumulated volume of permeate were checked every 15 minutes, while for each 500 mL of permeate accumulated, a sample was collected for COD analysis. At the end of the test, permeate and concentrate samples were collected to be characterized by COD concentration, total solids and color.

Scale up

First, the critical flux was measured using the TMP-step method (Field et al. 1995). For this, the membrane module, previously chemically cleaned, was submerged into the effluent and the permeate flux was monitored at fixed values of pressure. For each value of pressure, the filtration time was 15 minutes, after which the pressure was increased by 0.05 bar. The critical pressure corresponded to the value in which there was a permeate flux reduction during the 15 minutes with constant pressure.

The continuous pilot plant operation was performed according to the preset ideal conditions. A time of 20 seconds of backwash (1.6 L.h⁻¹) at every 22 minutes of filtration was applied. The permeate flux was continuously monitored for 1,000 hours. Based on the flux data, the effluent permeability was calculated. Over that period of time permeate and concentrate were periodically collected to be analyzed.

Economic aspect

The investment and operation and maintenance (O&M) costs of the concentration of vinasse by microfiltration was based on a system with treatment capacity of 1,000 m³.h⁻¹ that will demand 45,500 m² of membrane (considering permeate flux of 22 L.h⁻¹.m⁻², as will be shown). Investment costs include direct costs such as equipment, instrumentation and control systems for chemical cleaning,
storage tanks, generators, transformers, pumps, piping, valves and others; and indirect costs are estimated based on direct costs and represents the amount spent on activities not related directly to the material and labor, the work of general facilities, such as engineering and supervision services, etc. The startup cost, which is the amount of capital required to start the operation, corresponding to 8% of fixed investment, was also considered as investment cost. The startup cost, which is the amount of capital required to start the operation, corresponding to 8% of fixed investment, was also considered as investment cost. The startup cost, which is the amount of capital required to start the operation, corresponding to 8% of fixed investment, was also considered as investment cost. The startup cost, which is the amount of capital required to start the operation, corresponding to 8% of fixed investment, was also considered as investment cost. 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The O&M cost include the labor force for plant operations (one engineer and four technicians/operators), energy consumption, chemicals for cleaning the membranes, maintenance of the units, and replacement of membrane modules. To calculate the spending on labor, payment of 13 annual salaries plus 70% corresponding to taxes and labor charges were considered. For the maintenance cost a value of 5% of the initial investment was considered associated with preventive and corrective maintenance of the units. As an estimate of costs of chemicals used for cleaning the membranes, a value of 2% of the initial investment was considered. The power consumption was estimated to be 28.7 kW. The depreciation time of the treatment plant was considered to be 15 years.

**RESULTS**

**Evaluation of the operating conditions**

The permeate flow rate profile was checked for a raw effluent (pH 4.76) and effluent with neutral pH (Figure 2(a)). A rapid decrease of the permeate flux was observed within the first few minutes of filtration due to the concentration polarization. The posterior gradual reduction was due to the cake formation over the membrane surface, until reaching a stable permeate flux about 18.5 and 12.5 L.h⁻¹.m⁻² at pH 4.7 and 7, respectively. The lower permeate flux observed for the neutral pH effluent may be due to more intense membrane pore obstruction in this condition.

The microfiltration allowed a complete suspended solid retention, as expected. It was noticed that neutralization influenced color and COD retention (Table 2). When the effluent had been neutralized, it showed higher color intensity and lower color removal efficiency. The vinasse residual color may be related to the presence of melanoidins and phenolic compounds with low molecular weight. The medium neutralization favors the repolymerization of the
colored compounds, which explains the higher color intensity after pH adjustment. These compounds may show higher fouling propensity, corroborating with the results found for the permeate flux decrement when effluent neutralization was carried out. In addition, the pH adjustment with CaO would introduce calcium ions (Ca$^{2+}$) in the feed that can contribute to inorganic fouling. According to Seidel & Elimelech (2002), biopolymers generally contain ionizable groups, such as SO$_4^{2-}$, CO$_3^{2-}$, PO$_4^{3-}$ and OH$^-$, which can easily neutralize inorganic materials, such as Ca$^{2+}$, Mg$^{2+}$, Al$^{3+}$ and Fe$^{3+}$, to form flakes which contribute to fouling. Such findings suggest that, if necessary, the neutralization should be done after effluent microfiltration, that is, only the permeate should be neutralized. Such procedure requires lower CaO consumption, besides providing lower membrane fouling potential, which will allow for a lower operating cost.

An improved air distribution in the submerged permeation module may provide a better hydrodynamic condition and then prevent and control membrane fouling. Most of submerged modules that operate with aeration have their aeration system installed on the bottom of the membrane tank. In this work, a new aeration system, consisting of diffusers positioned among the membrane permeation fibers, was developed. Such better air distribution allows for a larger contact area between the air bubbles and the membrane surface and provides higher shearing efficiency.

A smaller fouling formation is noticed when the aeration is applied among membrane fibers (Figure 2(b)), which provided 63% higher permeate flux than when aeration system was located at the bottom of the tank. Aeration system has not influenced MF performance regarding organic matter and color retention (Table 3). A slight difference may be noticed regarding the total dissolved solid retention. In this case, the cake layer, formed over the membrane surface, behaves as a dynamic membrane, and helps to increase the rejection rate of solutes by the membrane.

With optimal operating conditions established, that is, feed with no pH adjustment and aeration among the membrane fibers, the recovery rate was then determined. The data obtained from vinasse concentration tests (Figure 3(a)) indicated that during operation with recovery rate ranging from 0 to 95% there was no significant change in membrane rejection of COD. The test showed that the membrane permeability reduction was only 54% even operating at very high recovery rate. Vasić et al. (2012) used 0.45 μm microfiltration membranes to concentrate vinasse, and observed a permeability reduction up to 55%, which is similar to the one found in this study, however with a recovery rate of 47%. This result suggests that a recovery rate of 93% could be applied, that is, the volume of vinasse to be managed could be reduced to 7% of the total volume.

During the vinasse concentration test it was observed, in a time of 400 h, that there was a decline in the permeate flux, causing a large variability in the effluent and water permeability rate ranging from 0.4 to 1.0. This may be caused by fouling in the membrane surface. To restore the permeate flux, the membrane was submitted to a chemical cleaning with sodium hypochlorite (200 mg.L$^{-1}$) during 20 min in an ultrasonic bath. After the cleaning procedure, no decrease in the permeate flux was observed until test finalization, suggesting that the new configuration with aeration between membrane fibers can provide the maintenance of permeate

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**Table 3 | Critical flux and membrane hydraulic permeability in pilot MF plant**

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Critical flux 1 (L.m$^{-2}$.h$^{-1}$)</td>
<td>13.1 (0.20 bar)</td>
</tr>
<tr>
<td>Critical flux 2 (L.m$^{-2}$.h$^{-1}$)</td>
<td>10 (0.30 bar)</td>
</tr>
<tr>
<td>Critical flux 3 (L.m$^{-2}$.h$^{-1}$)</td>
<td>8.1 (0.15 bar)</td>
</tr>
<tr>
<td>K$^a$ new membranes</td>
<td>300 (L.m$^{-2}$.h$^{-1}$.bar$^{-1}$)</td>
</tr>
<tr>
<td>K$^a$ fouled membranes</td>
<td>128 (L.m$^{-2}$.h$^{-1}$.bar$^{-1}$)</td>
</tr>
<tr>
<td>K$^a$ membranes after chemical cleaning</td>
<td>305 (L.m$^{-2}$.h$^{-1}$.bar$^{-1}$)</td>
</tr>
</tbody>
</table>

K$^a$ – hydraulic permeability.

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**Figure 3 | (a) Relationship between recovery rate, permeate flux and COD of permeate; (b) permeate flux and effluent and water permeability rate (K$\text{water}$/K$\text{water}$).**
flux during long periods of time. It can contribute to reduce the necessity of periodical chemical cleanings and the costs associated with chemical reagent and energy during microfiltration process.

Scale up

The critical flux for the system was analyzed for MF operating under the conditions previously determined, that is aeration (0.5 m$^3$.h$^{-1}$) among the membrane fibers and backwash flow rate of 1.6 L.h$^{-1}$ (Table 3).

The critical flux varied from 13 L.h$^{-1}$.m$^{-2}$ to 8 L.h$^{-1}$.m$^{-2}$, and this decrement may be related to changes in the vinasse characteristics, such as the concentration of solids and substances with higher fouling potential, for example melanoidins and phenolic compounds. It is worthy pointing out that these changes are common in industrial wastewater. In addition, one of the advantages of a pilot plant operation that receives real industrial wastewater is mainly the fact that system behavior can be observed as it undergoes real oscillations. Based on the critical flux results, the pressure of 0.10 bar was defined as the most suitable for continuous system operation, which was defined as the value right below the critical pressure value of 0.15 bar, set for the operation in a subcritical condition.

A chemical cleaning procedure using a solution of sodium hypochlorite at 250 ppm for 2 hours was performed after 20 days of continuous permeation. The membrane permeability with water before and after the cleaning procedure was measured (Table 3). A permeability reduction of 57% was found during the filtration time, which is explained by the pore clogging and adsorption of compounds on the pore walls and membrane surface. However, such fouling formation is reversible as the permeability may be fully recovered by the cleaning procedure.

Also observed was the value of decrease rate of main permeate flux of $-0.15$ L.h$^{-1}$.m$^{-2}$ per hour of filtration over the monitoring period (Figure 3(b)). It was noticed that the effluent and water permeability rate decreased slowly during the monitoring period. These results reinforce the low potential fouling of vinasse.

Potential applications of concentrated vinasse

Table 4 shows the physicochemical properties of permeate and concentrate obtained in bench scale operation under the optimal operating conditions (feed with no pH adjustment, aeration among the membrane fibers, and 93% of recovery rate). These findings show that concentrate keeps its usage potential in fertigation procedures, however with a low final volume to be discharged, with a reduction of 93%. The low ion retention was expected as the microfiltration process is not designed to retain such compounds. Nevertheless, the vinasse application on the soil is currently limited to the potassium concentration. Therefore the low potassium retention along with a high organic material retention allows the product to be used as an organic fertilizer. According to Guchert & Roussenq Neto (2007), organic material has a direct effect on the physical, chemical and biological soil properties. From the physical point of view, organic material contained in the soil improves its structure, reduces its plasticity and cohesion potential, and increases its water retention capacity and aeration.

High concentration of organic material and significant inorganic nutrients concentration also provides the concentrated vinasse a potential application as an organic compound supplement for bioremediation of contaminated soils, microbial biomass production, or substrate for biosurfactant production.

The use of vinasse has been evaluated as a source of nutrients for microorganisms in the decontamination of

![Table 4](https://iwaponline.com/wst/article-pdf/73/6/1434/462976/wst073061434.pdf)
organic residues present in the soil (Crivelaro et al. 2010). The utilization of vinasse effluent for microbial biomass production has been reported by several researchers (Barker et al. 1982). Dubey & Juwarkar (2001) demonstrated the production of an effective biosurfactant from Pseudomonas aeruginosa strain BS2 using vinasse effluent as a substitute for nonrenewable resources.

Regarding the agricultural methods of vinasse application in the soil, some environmental and irrigation parameters must be evaluated in further studies, such as soil type, soil field capacity (water retention) and the minimum concentration of salts that can be disposed in the soil.

The permeate produced as a byproduct of vinasse microfiltration may be used as water replacement in cases where the water quality standard is less demanding as, for example, for washing sugarcane before being ground for ethanol production, or may be spared for later purification treatment for more demanding uses.

Economic aspects

Table 5 shows the total costs of vinasse concentration by MF. As can be seen, the cost of vinasse microfiltration is equivalent to US$0.48 m⁻³ of vinasse.

The energy cost is rather significant, and corresponds to approximately 30% of the total operating cost, followed by cost of membrane replacements (20%), and preventive and corrective maintenance of the system (18%). Although power consumption is high, it is worthy pointing out that compared with evaporation traditionally used for vinasse concentration, this cost is significantly lower (Rausch & Belyea 2006).

<table>
<thead>
<tr>
<th>Items</th>
<th>Values (US$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total investment–CAPEX</td>
<td>11,606,465.00</td>
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<tr>
<td>Installed system cost</td>
<td>10,746,727.00</td>
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<tr>
<td>Startup cost</td>
<td>859,738.00</td>
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<tr>
<td>O&amp;M total–OPEX</td>
<td>3,332,226.00</td>
</tr>
<tr>
<td>Hand labor</td>
<td>290,000.00</td>
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<tr>
<td>Membrane replacement</td>
<td>856,000.00</td>
</tr>
<tr>
<td>Energy</td>
<td>1,239,021.00</td>
</tr>
<tr>
<td>Fund form maintenance of units</td>
<td>752,271.00</td>
</tr>
<tr>
<td>Membrane chemical cleaning</td>
<td>214,934.00</td>
</tr>
<tr>
<td>Total annual cost</td>
<td>4,105,990.00</td>
</tr>
<tr>
<td>Treatment cost (US$/m³ of vinasse)</td>
<td>0.48</td>
</tr>
</tbody>
</table>

CONCLUSIONS

The microfiltration process has been proved to improve vinasse value, and may offer an alternative to make the alcohol production more sustainable. The vinasse concentrate has high organic content concentration (organic fertilizer) and inorganic nutrients, and thus could potentiality be used for fertigation. It may also be used as an organic matter supplement for contaminated soil bioremediation or to produce microbial biomass or substrate for biosurfactant production. The permeate may be used as recycled water in cases in which the water quality standard is less demanding, or may be later treated to supply purified water demands.

REFERENCES


ANA 2005 Manual de Conservação e Reuso de Água na Agroindústria Sacroenergética. Agência Nacional de Águas; Federação das Indústrias do Estado de São Paulo; União da Indústria da Cana-de-açúcar; Centro de Tecnologia Canavieira. Brasília.


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