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Clarification of raw rice wine by ceramic microfiltration membranes and membrane fouling analysis

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Ceramic membrane capacity during the filtration of raw rice wine is typically limited by fouling, which can occur by pore blocking, pore constriction or caking. In this study three modified fouling models were used to describe flux decline behavior during microfiltration accounting for these three classical fouling mechanisms. The fouling mechanism was identified by estimation of the model parameter according to a nonlinear regression optimization procedure. Analysis by the models indicated cake filtration to be the dominant mechanism and pore constriction to be the secondary fouling mechanism. In the fixed operating conditions of transmembrane pressure (TMP) and cross-flow velocity (CVF), the fouling mechanism evolves from a pore constriction to a cake filtration. Membrane fouling was also investigated using Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM). FTIR analysis revealed that polysaccharides, long chain protein and some phenolics appear to be primarily responsible for fouling. The main physico-chemical characteristics of rice wine were evaluated in order to select membrane pore size and material that supply the highest permeation flux and best clarified rice wine.

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1. Introduction

Rice wine (RW) is one of the world's most ancient wines [1]. Produced for thousands of years, the unique brewing technology handed down for generations gives RW its characteristic bright brown color, subtle sweet flavor, and low alcohol content. In China, RW has been popular for centuries and has the honor of being the national banquet wine [2]. RW is also widely known for its health enhancing properties. It is not only a good tonic, but is also frequently used in Chinese traditional medicine as a therapeutic component. RW has also been claimed to have beneficial effects for the prevention of cancer and cardiovascular disease; these benefits are related to its *in vivo* antioxidant activity [3–5]. Because of its unique characteristics, RW offers advantages for specific health conditions and, consequently, it has a great potential for industrial exploitation.

Traditionally, RW is brewed from glutinous rice and wheat. Glutinous rice contains higher protein and lower fat than wheat, while the wheat provides abundant carbon, nitrogen, and microelements for the molds and yeasts used for fermentation. Following fermentation, raw RW contains substantial amounts of bacteria, enzymes, protein, polyphenols, iron ions, and pentosans, all of which can affect the non-biological stability and characteristics of RW unless further processing is carried out [6].

The main goal of RW processing is foremost to obtain a safe and stable product. In order to guarantee the non-biological stability, RW is industrially pasteurized at 85 to 90 °C for 30 min, then stored in a tank for diatomite filtration for sediment removal. These processes increase the shelf life of the product and assure its safety, but they also affect the sensory properties of the RW, which depends on volatile substances that are largely heat sensitive [7]. Moreover, the sediment is difficult to filter out using traditional methods. Its removal from RW after pasteurization entails a loss of approximately 10% of the total volume of the brewed product [8].

Cross-flow microfiltration has become extensively used in the winemaking industry in recent years, having proven to be a useful clarification and microbiological stabilization technique [9]. Its increasing popularity is largely attributed to the fact that the membrane separation process is a gentle one that involves no phase changes or chemical agents [10]. Consequently, the introduction of this type of technology into the industrial production of the RW may represent one of the answers to the problem of producing RW that is of high quality, naturally fresh tasting, and additive-free.

Ceramic membrane filtration is an advanced method for separating unwanted substances from fermentation liquids. Due to its superior selectivity, permeability, and thermal and chemical stability over conventional processing methods [11], ceramic membrane filtration has potential application in the clarification of RW. Its advantages include: improvement

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in RW yield; the possibility a single step operation that would reduce working times; the possibility of avoiding the use of gelatins, adsorbents, and other filtration aids; the reduction in enzyme utilization; ease of cleaning and maintenance of the equipment; reduction in amounts of waste products; and elimination of the need for pasteurization [12,13]. P.Y. Tan et al. [12] , D.Y. Liu et al. [13] and J.X. Huang et al. [14] have studied ceramic membranes and the influences of operating conditions and membrane pore size on the flux and the physico-chemical characteristics of feed and permeation during microfiltration of raw RW. They were able to obtain suitable operating conditions and proved that this new technology is feasible for RW clarification.

However, the permeation flux was found to decrease dramatically during the filtration process, due to the adsorption of solutes on the outer membrane and the inner pore surfaces. This effectively blocks the pores with the excluded solutes and forms a thick cake layer of precipitated solutes on the membrane surface [12–16]. This membrane clogging or fouling reduces productivity, and also can potentially shorten membrane life. Minimization of membrane fouling is therefore essential for the membrane process to be economical. In order to minimize fouling, a detailed understanding of the causes of flux decline and the relative contribution of each cause is needed. For example, if adsorption is the main cause of fouling, then improvement of mass transfer by altering membrane hydrodynamics would be of little benefit [17].

To understand the mechanisms of flux decline, some researchers have proposed a resistance-in-series model to analyze the fouling mechanism [18–20]. Based on this model, S.T.D. de Barros et al. [21] analyzed the fouling mechanism during pineapple juice clarification. Their analysis revealed that a hollow fiber membrane separation process is controlled by a cake filtration mechanism, while a pore blocking fouling mechanism controls a ceramic tubular membrane. A. Cassano et al. [22] described the permeation flux decline observed during the ultrafiltration (UF) of blood orange juice, examining operation effects and fluid-dynamic parameters on permeation fluxes. N.K. Saha et al. [23], who studied polysaccharides fouling during sugarcane juice UF, found cake fouling to be the dominant mechanism. Overall, the model is suitable for analysis of the fouling mechanisms in microfiltration of many food products, bioprocessing solutions, fruit juice, and pharmaceuticals. However, lack of research into fouling behaviors limits the wide usage of ceramic microfiltration membranes in raw RW sterilization processes in China.

In the present study, a modified model of the differential equation used to describe classical dead-end filtration was applied to describe the flux decline observed during microfiltration of raw RW through ceramic membranes. Membrane fouling was also investigated using Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM). The aim of this study was to provide fundamental information regarding the control of membrane fouling during MF of raw RW.

2. Materials and methods

2.1. Membranes and raw rice wine

Three ceramic tubular microfiltration membranes with varied nominal pore sizes and different materials (Nanjing Jiusi High-Tech Co., Ltd, Jiangsu, PR China) were selected for use in this experiment. The characteristics of the ceramic membranes are presented in Table 1. The study used raw rice wine sampled from a local food plant (Huaian food plant, Jiangsu, PR China). Some physico-chemical characteristics of the raw rice wine are presented in Table 2.

2.2. Experimental set-up and procedure

Fig. 1 illustrates a schematic diagram of the cross-flow microfiltration set-up. The feed, in the amount of 2.5–3 L, was recycled between the retentate reservoir and the module by a rotary pump and the feed flow rate was controlled with a laboratory-constructed rotameter. TMP was controlled by the in/outlet valves. The TMP was calculated as the mean value of inlet and outlet pressure. The permeate was collected in a reservoir placed on a digital balance that could be connected to a computer. In a total recycle experiment, both permeate and concentrate flow were returned to the feed tank to keep concentrations constant.

For all experiments, the system temperature was fixed at 15 ± 3 °C to avoid sedimentation or affecting the characteristics of the rice wine. The TMPs were 0.10, 0.15, 0.18, and 0.20 MPa. The CFVs were 0.73, 1.10, 1.47, and 1.98 m s^{-1}.

After each experiment, the membrane was cleaned for the next use. The cleaning process varied according to the feed property. For this feed (Table 2), the membranes underwent a cleaning process utilizing 2%(w/w) NaOH and 0.15 M HNO_3 aqueous solutions at 40 ± 3 °C with a final rinse with water until the original permeation flux was restored [22]. The membranes were stored in NaOCl (preservative) to prevent bacterial formation [23].

2.3. Analysis

The feed and permeate were analyzed for turbidity, total solids, total insoluble solids, protein, dextrin, absorbance, and the content of target composition (such as ethanol, total acid, total polyphenols, amino acid nitrogen, and reducing sugars). Turbidity was measured with a 2100 Turbidimeter (HACH USA) and absorbance with a spectrophotometer (722, Shanghai, PR China) at 420 nm. Total insoluble solids, dextrin, ethanol, total acid, total polyphenol, amino acid nitrogen, and reducing sugars were examined by the Huaian food plant according to National Standard of the People's Republic of China (GB/T13662-2000).

The SEM analysis was done with a JSM-6300 scanning electron microscope (JEOL, Japan). Prior to SEM analysis, the membrane specimens were carefully taken from the middle of the elements (lengthwise) using a pair of tweezers and were sputter coated with gold. Information about the presence of specific functional groups on

Table 1
Characteristics of membranes.

Characteristics	Membrane 1	Membrane 2	Membrane 3
Material	α-Al_2O_3	α-Al_2O_3	ZrO_2
Nominal pore size (nm)	200	500	200
Membrane thickness (μm)	15	15	15
Membrane area (m^2)	0.00328	0.00328	0.00328
Inside diameter (ID) (mm)	7	7	7
Membrane length (cm)	15	15	15

Table 2
Physico-chemical characteristics of raw rice wine in its feed and permeate clarified by ceramic membrane with various nominal pore sizes and materials (TMP = 0.10 MPa, T = 15 ± 3 °C, CFV = 1.10 m s^{-1}).

Parameters	Feed	Permeate (200 nm α-Al_2O_3)	Permeate (500 nm α-Al_2O_3)	Permeate (200 nm ZrO_2)
Ethanol (v/v%)	18.2	17.8	18.0	17.7
Total acid (g L^{-1})	5.2	4.9	5.1	5.0
Total insoluble solids (g L^{-1})	12.7	0.8	1.2	0.7
Reducing sugars (g L^{-1})	17.6	17.2	17.4	17.0
Amino acid nitrogen (g L^{-1})	0.85	0.80	0.82	0.81
Crude protein (g L^{-1})	10.8	2.78	4.32	2.56
Total polyphenol (g L^{-1})	10.68	7.76	8.68	7.68
Absorbance at 420 nm	0.269	0.165	0.182	0.166
Dextrin (g L^{-1})	1.342	1.211	1.251	1.210
Turbidity (NTU)	35.3	2.13	3.10	2.12
Sediment (yes or no)	Yes	No	No	No
Steady flux (L m^{-2} h^{-1})		23	18	33

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