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Advances in the Precoat Filtration Process

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Abstract: Precoat filtration with body-feed is widely applied during wine-making. This technique employs filter aids, such as diatomites (silica particles), to form the precoat layer. However, the spent diatomites, which retain organic matter, represent an important source of pollution and land disposal of this type of waste is forbidden. In addition, these spent diatomites are not regenerable. The aim of this study was to evaluate regenerable filter aids which have a high environmental acceptability. Polyamide particles were characterized as filter aids, tested in precoat filtration and compared to diatomites which are the classic filter aids used in wine filtration.

Polyamide particles seemed to be efficient filter aids, reducing turbidity by 72% and the fouling index by 56% without affecting wine quality.

Keywords: Environment, filter aids, precoat filtration, wine

INTRODUCTION

Among classic filtration techniques, precoat filtration is one of the most effective in the clarification of highly fouling products such as gelatine, beer or wine. Filter aids, the particles that form the precoat layer, due to their complex shape and surface properties, are able to capture very fine particles as well as macromolecules. Precoat filtration, largely used in wine production, is often carried out using diatomites (silica particles) as filter aids. To properly maintain the filter aid precoat layer, and to maintain

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porosity, body-feed of filter aids can be used to supplement the unfiltered suspension. The filter aids are added to the feed and, together with the suspended solids from the raw liquid, build up a porous filter cake (1). The optimum amount of filter aid used depends on the size, the amount and the type of suspended solids to be removed. In wine production, the amount of filter aid usually applied is about 1 kg of filter aid per m^2 of filtering surface for the precoat layer formation and 1 g/L of solution to be filtered, for the body-feed.

Unfortunately, the spent filter cake represents an important source of pollution and the land disposal of this waste is forbidden. The spent filter cake is usually spread on the ground or destroyed, which generates environmental pollution and additional costs. The spent diatomites regeneration is not efficient. Spent diatomite recycling consists in calcination at 780°C. Although the recycled diatomites are of good quality, they are of no particular grade. This limits the possible recycling of diatomites to a maximum of 40% in breweries (2). New trends in processing are focused on environmental protection. The aim of this study was to test new materials as filter aids in order to replace diatomites in wine filtration. Polyamide particles were selected and tested as new filter aids because they are available in different sizes and are compatible with the equipment and the product to be filtered. Polyamide particles have already been tested in the brewery industry (3). Precoat filtration tests were carried out and the efficiency of the new filtering particles was determined with regard to turbidity reduction and fouling index improvement. The impact of new filter aids was tested using microbiological tests, and chromatic and chemical analyses of enriched red wine filtered on the polyamide particle precoat layer.

MATERIALS AND METHODS

Tested Filtering Particles

The classic filter aids used in comparison with the new polyamide filtering particles were Diatomyl® P00 (D_{P00}) employed for coarse filtration. These diatomites were provided by Laffort Œnologie. The polyamide particles (PAP) Rilsan®, used in this study, are available in 3 sizes Rilsan T (PAP_T), MC (PAP_{MC}) and ES (PAP_{ES}) and were provided by Arkema. The characteristics of these particles, measured in a previous study (4), are presented in Table 1.

Physical Measures of Filtrates

The filtration efficiency was estimated by turbidity (Turbidimeter HACH®) and fouling index. The fouling index is represented by the filtered volume

Table 1. Tested particles characteristics

Particles	Nature	Size (μm)	Specific surface area (m^2/g)	Water permeability (Darcy)
D_{P00}	Silica powder	15	0.85	4.46
PAP_{ES}	Polyamide powder	35	0.55	0.81
PAP_{MC}		50	0.43	3.5
PAP_T		110	0.16	9.27

obtained in five minutes (named V_5) at 2.10^5 Pa through a Millipore® membrane (diameter of 2.5 mm, area of 3.9 cm^2, and pore size of 0.65 μm) (5). When filtration was efficient, turbidity was low and V_5 was increased.

Chemical Measures of Filtrates

Chemical analyses of the filtrates were carried out to determine total sulphur dioxide (total SO_2), free sulphur dioxide (free SO_2), titratable acidity (TA), volatile acidity (VA), the alcohol content by volume, and pH. Total and free SO_2 were determined using the Ripper method (6, 7). The TA was determined by titrating 5 ml of decarbonised sample (CO_2 removed) and adding to dibromo-3′,3-thymolsulfonephtaleine (BBT) with 0.1 N NaOH and was expressed as g tartaric acid/L. For VA, a 15 ml sample was decarbonized, then a distillation system (CHENARD SPINOSA) was used to transform volatile substances from the sample into vapor by heating. The condensation from this vapor resulted in a distillate which was titrated by the Duclaux-Gayon method. VA was expressed as g acetic acid/L. An ebulliometer (BARUS–SEPCA) was used to determine the alcohol content of the sample at boiling point, and pH was measured with a pH-meter (pH 538 Multical® WTW) standardized to pH 7.0 and 4.0.

Chromatic Measures of Filtrates

The chromatic characteristics of the filtered wine measured were: total phenolic compounds (I index), total tannins, color intensity of the sample (CI and CI′), total color (TC), contribution of each color, and the red aspect. Therefore absorbencies were measured at 420 nm, 520 nm, 550 nm, 620 nm, and 280 nm using an UV/VIS spectrophotometer (V-530 JASCO). For the I index, 1 ml of sample was diluted in 100 ml of distilled water and the absorbance obtained at 280 nm was multiplied by a dilution factor of 100. Total tannins were determined using 2 glass tubes containing 4 ml of sample, 2 ml of distilled water and 6 ml of *12 N* HCl. One tube was placed in a heater (100°C for 30 minutes), and 1 ml of ethanol was added to each tube and the absorbance at 550 nm was then measured. The tannin concentration (C_T) was then read on calibration curve.

$$C_T(g/L) = 19.33 \times \Delta d_{550} \tag{1}$$

CI was calculated by adding absorbencies at 420 nm and 520 nm and CI′ was calculated by adding CI and absorbance at 620 nm. TC was calculated by dividing absorbance at 420 nm by absorbance at 520 nm. The contribution of each color was determined by calculating the proportion of CI' at each absorbance (420 nm, 520 nm, and 620 nm), corresponding to yellow, red, and violet, respectively. The red aspect (red spectrum) (d.A%) was calculated from the formula:

$$d \cdot A = \left[1 - \frac{d_{420} + d_{620}}{2 \cdot d_{520}}\right] \times 100 \tag{2}$$

where d_{420}, d_{520}, and d_{620} were absorbencies at 420, 520, and 620 nm, respectively.

Microbiological Analyses

Yeast strains, acetic bacteria, and lactic bacteria were enumerated from filtered wine samples following the enological codex rules (OIV, 2007). Yeast strains were cultivated at 25°C in 10 ml of solid YPG medium (yeast extract 10 g/l, bactopeptone 10 g/l, glucose 20 g/l, pH adjusted to 4.0 with *10 M* NaOH) supplemented with antibiotics (diphenyl (75 mg/10 ml ethanol) 2% and penicillin (12.5 mg/10 ml of sterile water) 1%). Bacterial strains were cultivated at 25°C in aerobic conditions for acetic bacteria and in anaerobic conditions for lactic bacteria, both in 10 ml of solid Dubois medium base (yeast extract, 5 g/l; neopeptone, 5 g/l; DL-malate, 10 g/l; $MgSO_4 \cdot 7H_2O$, 50 mg/l; $MnSO_4 \cdot H_2O$, 20 mg/l; centrifuged tomato juice (10,000 g, 10 min) 25% (v/v); pH adjusted to 4.5 with *10 M* NaOH) supplemented with pimaricine (50 mg/10 ml of sterile water), 2% (v/v) for lactic bacteria counts and with 1% (v/v) of penicillin (12.5 mg/10 ml of sterile water) and pimaricine (50 mg/10 ml of sterile water) 2% (v/v) for acetic bacteria counts.

EXPERIMENTAL SET-UP

The filter (Eurofiltec®) allows precoat filtration at laboratory scale, i.e., smaller volumes of sample. It is composed of a tank ("tank 1") for the

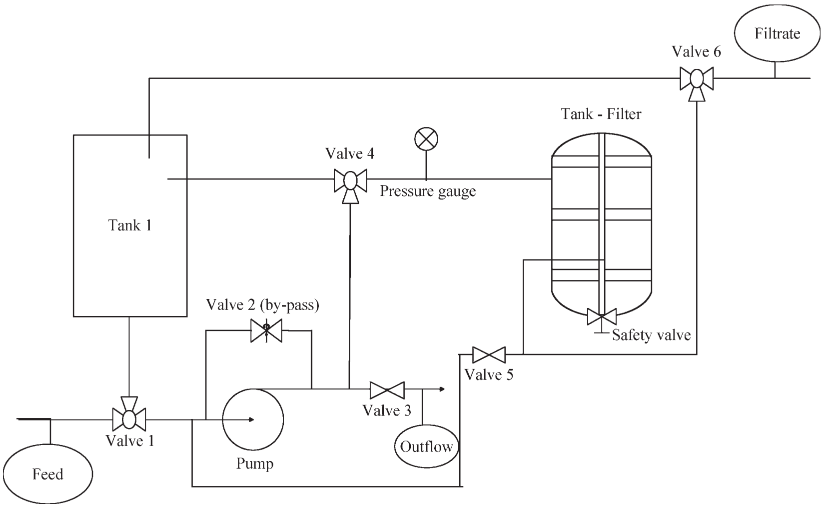

Figure 1. Precoat filter diagram.

suspension to be filtered and for the filter aid deposits (to form the precoat layer and/or for body-feed), a tank with 3 woven cloth filter elements and a centrifugal pump with a bypass (Figs. 1 & 2). The filtering surface was 0.024 m^2.

Fifty grams of filter aid were deposited in the closed loop during 30 minutes in order to ensure the perfect formation of the precoat layer on the filtering surface (2 kg/m^2). For precoat filtration tests, 7.5 L of suspension were filtered through the precoat layer (without body-feed) at 5.10^4 Pa. In this experiment, the filtration flux was not measured.

Figure 2. Precoat filter.

Table 2. Composition of enriched wine

Red wine with acetic bacteria	2.3 L
Yeasts	20 mL (10^3 UFC/mL)
Aromas:	
Chloroanisoles	20 ng/L
Whisky lactone	400 μg/L
Geosmin	200 ng/L
Red wine	qsp 35 L

Suspension Composition

A synthetic solution was set up in order to test the media. The tested suspension composition is shown in Table 2.

Chloroanisoles can impart "musty" or "mouldy" off-flavors to wine, other beverages, and foods. The most common chloroanisole found in wine is TCA, which is the common abbreviation for the chemical compound 2,4,6-trichloroanisole, the first source identified and thought to be the primary cause of "corked taste." Other chloroanisole contaminants of wine may include 2,3,4,6-tetrachloroanisole (TeCA), pentachloroanisole (PCA) and 2,4,6-tribromoanisole (TBA). Geosmin (trans-1,10-dimethyl-trans-9-decalol) is the compound responsible for the distinctive "earthy" odor that resembles "mustiness" in wine. Chloroanisoles and geosmin ruin wine aromas. In contrast, whisky lactone (β-methyl-Y-octalactone), due to woody compounds, is responsible for the high quality of alcoholic beverages (whisky, wine, brandy, and Scotch) and has a coconut-like smell. All of these aromatic molecules were added to determine the impact of precoat filtration on the organoleptic qualities of filtered wine. These molecules were identified by gas chromatography-mass spectrometry (GC-MS).

RESULTS

The suspension could be compared to aged wines which are usually clarified in filter presses or precoat filters using coarse diatomites. The filtrates were recovered for analyses and the results of these analyses are shown in Table 3.

All filter aids tested enhanced filtration efficiency. Concerning turbidity removal (Fig. 3), the filtration efficiencies were comparable for D_{P00}, PAP_T, and PAP_{MC}. With regard to D_{P00} and PAP_T filtrations, the filtrates were of the same quality. PAP_T had filtration characteristics comparable with those of D_{P00}. The results obtained with PAP_T suggested that these particles can be used as a coarse filter aid. The low V_5 improvement efficiency of these particles could be explained by the large precoat layer, which allowed small particles or micro-organisms to pass through the filter cake. These particles

Table 3. Physical and chemical analyses of red wine filtrates obtained by precoat filtration at $5 \cdot 10^4$ Pa

	Control (no filtered)	D_{P00}	PAP_T	PAP_{MC}	PAP_{ES}
Physical measures					
Turbidity (NTU)	20 ± 2	9.8 ± 0.6	10.3 ± 0.7	13.6 ± 1.5	5.5 ± 0.6
V_5 (filtered volume in 5 minutes)	72	117	87	165	156
Chemical analyses					
Titratable acidity (g/L tartaric acid)	5.5	5.5	5.4	5.1	5.2
Volatile acidity (g/L acetic acid)	1.29	1.28	1.27	1.20	1.22
Alcohol content by volume (%)	11.7	11.6	11.7	11.6	11.5
pH	3.4	3.5	3.5	3.5	3.5
Free SO_2 (mg/L)	26	26	26	26	24
Total SO_2 (mg/L)	72	68	72	68	70
Color control					
Total polyphenol index	53.4	48.2	52.9	51.9	50
Total tannins (g/L)	3.4	3.0	3.0	3.0	2.9
Color intensity (CI)	0.67	0.62	0.64	0.63	0.62
Color intensity 2 (CI′)	0.76	0.71	0.73	0.71	0.70
Yellow (d420%)	39.6	39.6	39.5	39.4	39.2
Red (d520%)	48.1	49.1	48.7	49.4	49.9
Violet (d620%)	12.3	11.3	11.9	11.2	10.9
Red aspect (%)	46.1	48.1	47.3	48.8	49.7
Microbiological tests					
Yeast (UFC/100 ml)	$2 \cdot 7\ 10^{+4}$	$3 \cdot 4\ 10^{+2}$	$1 \cdot 6\ 10^{+4}$	50	$2 \cdot 9\ 10^{+2}$
Total bacteria (UFC/ml)	$2 \cdot 5\ 10^{+5}$	$1 \cdot 3\ 10^{+5}$	$2 \cdot 9\ 10^{+4}$	$2 \cdot 3\ 10^{+4}$	$2 \cdot 3\ 10^{+4}$

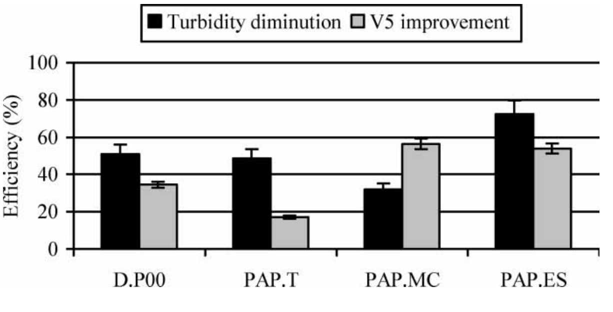

Figure 3. Precoat filtration efficiencies.

can thus be used to carry out coarse filtrations or clarifications. PAP_{ES} may be used for fine filtration as turbidity reduction was more than 75%. Polyamide particles (ES & MC) were the most interesting filter aids with regard to fouling index improvement (V_5). Filtration efficiencies and microbiological test results showed that PAP_{MC} could be used for fine filtrations and PAP_{ES} for even finer filtrations or pre-bottling filtrations. The application of these filter aids during various stages of wine-making is only possible if the new filter aids are used in adapted equipment.

The differences in the chemical analyses of wine following filtration with the new filter aids were not significant (Fig. 4). None of the tested particles seemed to change the chemical characteristics of filtered wine. The free SO_2 with PAP_{ES} was lower than with the other particles, however, the Ripper method used to measure SO_2 was slightly inaccurate.

Another important property of wine is its color. Wine color is due to polyphenolic and anthocyanin compounds which are fragile. Polyphenols are substances which are present in the colouring matter and tannins of wines. Tannins are organic compounds characterized by their astringent taste. The measurement of chromatic characteristics reflected the polyphenolic and anthocyanin composition of the filtrate.

Red wine filtration tests using diatomites carried out by Serrano and Paetzold (8), showed that only fine filter aids had an effect on the chemical composition which was caused by a reduction in tannins and anthocyanes of about 10%. This was also observed in this study, where a reduction in tannins of 12.5% with PAP_{ES} and 11% for the other filter aids (D_{P00}, PAP_T, and PAP_{MC}) were observed (Fig. 5). Filtration on polyamide particle precoat layer, even on PAP_{ES}, did not greatly affect polyphenols (IPT)

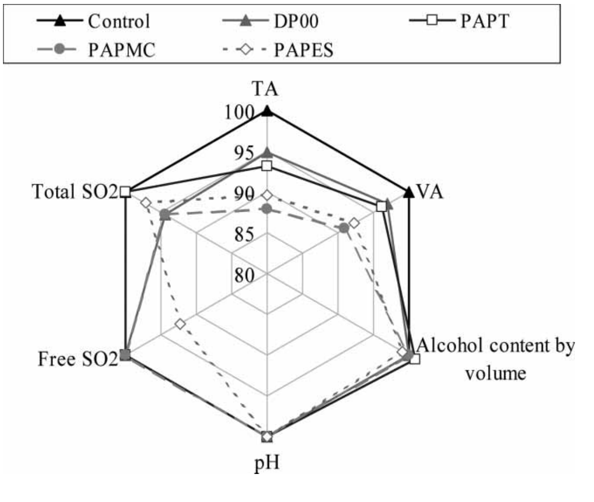

Figure 4. Filtrates chemical modifications (in comparison with unfiltered red wine).

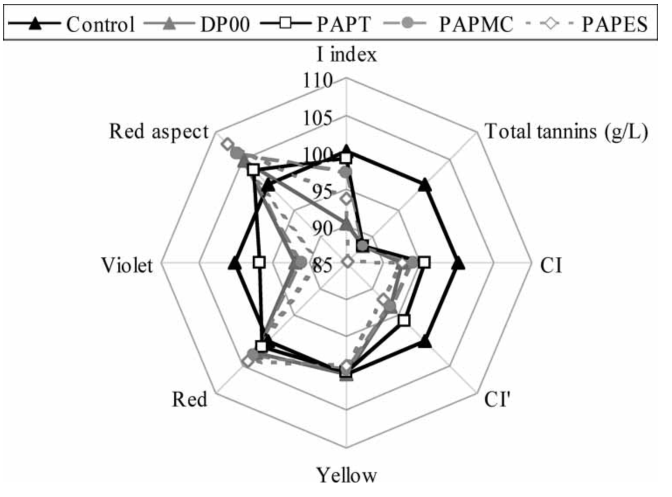

Figure 5. Filtrates chromatic modifications (in comparison with unfiltered red wine).

compared with filtration on D_{P00} (Fig. 5). Chromatic characteristics of each filtered wine were not significantly different. Red aspect represents brilliance of the wine measured using the red spectrum. As the filtration process decreased DO_{420} and $DO_{620,}$ corresponding to yellow and violet, DO_{520} (red color), as well as the red aspect, appeared to increase.

The particles of PAP_{MC} and PAP_{ES} seemed to retain more yeasts than D_{P00}. This was not the case for PAP_T whose pores in the precoat layer were larger (9). On the other hand, all the polyamide particles retained more bacteria that $D_{P00,}$ which was related to the electric charge of the polyamide particles.

Among chloroanisole molecules, only TCA and PCA were measured in both control and filtrates (Fig. 6). TeCA and TBA were not detected. The chloroanisole content was similarly slightly decreased during filtrations on D_{P00} and PAP_T. Chloroanisoles were retained more on PAP_{MC}. The results

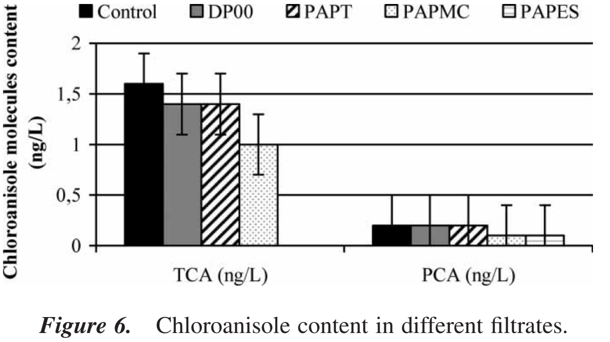

Figure 6. Chloroanisole content in different filtrates.

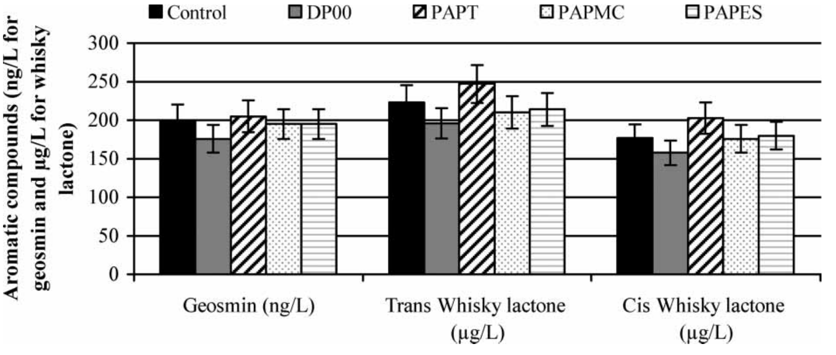

Figure 7. Geosmin ("earthy-musty") and whisky lactone ("coconut") rate in different filtrates.

obtained for PAP_{ES} filtration were not accurate. In conclusion, there were no significant differences in aroma content between filtrate samples.

Other flavors were retained slightly by D_{P00} filtration. Indeed, geosmin was decreased by 12%, the trans and cis whisky lactones were retained by a total value of 11% and 13%, respectively. PAP_{MC} retained 2% of geosmin, 6% of trans whisky lactone and only 0.5% of cis whisky lactone. PAP_{ES} retained 2% of geosmin and cis whisky lactone and 4% of trans whisky lactone. PAP_T did not retain any of these flavors (Fig. 7).

Wine quality is the most significant criteria with regard to filtration performance. In order to determine the differences in organoleptic characteristics, two types of sensorial analyses were carried out: a preference test and triangular tasting (data not shown, (4)). Tasting did not reveal any particular distinctions between the samples. Diatomites are felt by tasters but no preferences were observed. However, the majority of tasters preferred the check sample. This may be explained by the fact that none of the particles had been cleaned before use and diatomites need to be rinsed to avoid the taste of paper or cardboard. Polyamide particles do not impact on the organoleptic characteristics of the wine and do not require rinsing before use.

CONCLUSIONS

Polyamide particles can replace diatomites as filter aids in wine filtration as they are available in various sizes, and have good filtration efficiency resulting in reduced turbidity and improved fouling index. PAP_T are similar to D_{P00} with regard to filtration efficiency. PAP_{ES} and PAP_{MC} are finer filter aids.

These tests also showed that the polyamide particles used do not affect the chemical parameters of the wine during filtration. The polyamide particles did not retain a significant amount of the polyphenols contained in wine. The results from both chemical analyses and chromatic characteristics of different wines in contact with each of the particles studied did not show any significant differences. The organoleptic qualities of the wine were only slightly modified by filtration on polyamide particles, and in some cases improved.

Polyamide particles are valorizable by incineration or spreading and are reusable. Some filtration tests have been carried out to determine the best method of cleaning Rilsan particles. These particles are regenerable by back-washing with water and air or with NaOH (1%) (10), they are also non-polluting for the end-user and the environment.

In addition to their filtration efficiency, polyamide particles are interesting because:

- waste quantity is reduced compared with spent diatomites, resulting in decreased soil contamination,
- the effluent can be discharged, resulting in decreased contamination of groundwater,
- incinerator loads will be limited, resulting in reduced volatile organic carbon (VOC) emission as well as ash (as compared to silica),
- health risks associated with the use of silica filter aids will be reduced.

Moreover, because of their interesting calorific value, the spent polyamide particles may be incinerated (pyrolysis), resulting in renewable energy production (heat and/or electricity). Pyrolysis was advocated by the European Union because it is more acceptable to human health and enhances environmental protection.

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