NEW FILTER AIDS FOR WINE FILTRATION

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Abstract

Aims: In a real concern to improve the quality and to reduce the pollution, the concept of « ecological filtration » based on the use of a regenerable filter aid is developed.

Methods and results: Polyamide particles were tested as new filter aids for wine filtration. The particles were characterized with regard to water permeability and specific surface area. Filtration experiments were carried out at pilot scale under industrial operating conditions. Two test runs were performed using different suspensions and three grades of polyamide particles.

Conclusions: In the first precoat filtration tests, the impact of polyamide particles on wine quality was determined by the chemical characteristics of the wine. Enological analyses showed that the precoat filtration of wine with polyamide particles did not affect wine quality and improved turbidity (97%), fouling index (80%) and the filtrate flux (compared to the usual precoat filtration with diatomites) . In the second test run, polyamide particle regeneration tests proved that these particles could be re-used as filter aids several times.

Significance and impact of study: Polyamide particles used as filter aids in wine filtration were found to be very effective. Moreover, these particles are available in several sizes allowing their application at different stages of the wine-making process.

Key words: filter aids, precoat filtration, polyamide particules, regeneration, wine

Résumé

Objectif : Dans un souci d'amélioration de la qualité de filtration et de la réduction de la pollution, le concept de « filtration écologique » basé sur l'utilisation d'un adjuvant de filtration régénérable est développé.

Méthode et résultats : Des particules de polyamide ont été testées comme nouvel adjuvant de filtration. Les particules ont été caractérisées en termes de perméabilité à l'eau et de surface spécifique Les expériences ont été réalisées sous des conditions de fonctionnement industriel. Deux tests ont été réalisés en utilisant des suspensions différentes et trois catégories de particules de polyamide.

Conclusions : Dans les premiers tests, l'impact des particules de polyamide sur la qualité de vin a été appréhendé en suivant plusieurs caractéristiques chimiques du vin. Les analyses ont montré que la filtration avec des particules de polyamides n'a pas affecté la qualité de vin et a amélioré la turbidité (97 %), a diminué l'indice de colmatage (80 %) et a augmenté le flux de filtrat (comparé à la filtration avec diatomites). Dans le deuxième test, les tests de régénération de particule ont prouvé que ces particules pourraient être réutilisées plusieurs fois.

Impact : Ces particules de polyamide utilisées comme adjuvants de filtration de vin sont très efficaces. De plus, elles sont disponibles dans plusieurs tailles, permettant ainsi leur application à différentes étapes de l'élaboration du vin.

Mots clés : adjuvant de filtration, filtration sur précouche, particules de polyamide, régéneration, vin

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INTRODUCTION

The beverage industry, like a large number of other industries (chemical, pharmaceutical, food processing, water treatment and waste water treatment), use filter aids for precoat filtration with or without body-feed. The precoat filters consist of a filter aid layer supported by a filter cloth (septum). To prolong filtration cycle time and to prevent premature cake plugging (reduce cake resistance), filter aids may be added to the unfiltered liquid known as the body-feed. The body aids and the solids from the raw liquid build up into a porous filter cake (Wakeman, 2007). Filter aids are inorganic mineral powders or organic fibrous materials, which are used to enhance filtration performance (Kamigasa and Sumi, 1993). Commonly encountered filter aids include diatomites, perlites and celluloses. Some of these materials have been in use for over 80 years (Sulpizio, 1999). The most economical and effective way to filter beer and wine is to use diatomites, which are the fossil remains of single-celled microscopic algae called diatoms, which consist of a porous silica skeleton. However, the crystalline silica contained within the diatomites is a health hazard, therefore safe working conditions are essential (Le Du *et al.*, 2001). The optimum filter aid and body-feed grade depends on the size and the type of suspended solids to be removed. In wine filtration, the size of the diatomites used as filter aids is usually between 2 and 100 µm. The precoat filtration velocity is about 0.3 to 0.5 $m^3/(m^2.h)$ for a liquid charged with clogging impurities (rough wine filtration or early clarification of wine lees or wine must) and 1.5 to 2.5 $m^3/(m^2.h)$ for a pre-treated wine (fine or sterile filtration). Generally, the applied pressure (the difference between dynamic pressure and static pressure) is between 5.10^4 and 6.10^5 Pa. The amount of filter aid usually applied is about 1 kg of filter/m^2 of filtering surface for the precoat layer and 1 g/L of solution to be filtered, for the body-feed.

After filtration, the spent filter cake is composed of diatomites mixed with raw wine impurities. Regeneration of diatomites is not really efficient as their particle size distribution and permeability are changed after regeneration, which limits their possible recycling (Hermia and Rahier, 1993). The spent filter cake is usually deposited on or into the ground, which generates environmental pollution. According to the French Environmental Directive N°646-92 as of July 2002, only ultimate waste disposal is allowed; thus disposal of the spent diatomites used in precoat filtration is a problem. In fact, the treatment, recovery and disposal of diatomite waste, is becoming increasingly problematic. Since diatomites are employed in several stages of the winemaking process (wine and must clarification, tartaric acid crystals removal, pre-bottling filtration, etc.), the amount of spent and contaminated diatomites in wine filtration is considerable (10,000 t/year in France).

With this in mind, the present study was conducted with the objective of finding other particles, which could be used as filter aids. The new particles used as filter aids had to be inert and available in different sizes due to their various applications in wine production. The particles also had to be easily cleaned, reusable and recyclable. Rilsan®, polyamide 11 particles (PAP), are fully polymerized thermoplastic particles and are produced from a regenerable and renewable raw material of plant origin, the Castor plant (*Ricinus communis*). PAP are usually used to coat pipes in water transportation equipment and water treatment plants to prevent corrosion. PAP are available as ready-to-use powders in various grades (fine to large) and are compatible with food products, drinking water and other fluids. Because of these properties, PAP have been chosen and tested as filter aids with the aim of replacing diatomites. The aim of the present study was to analyze the filtration characteristics of PAP and to compare their performance with those of the usual enological filter aid, diatomites. Precoat filtration tests with and without body-feed were performed with diatomites and with PAP. To characterize the performance of the filter aids, water permeability and capture efficiency (decreased turbidity and improved fouling index) were measured. The organoleptic impact of the filter aids on wine was evaluated by means of chemical and physical analyses of filtrates.

MATERIALS AND METHODS

The tested diatomites were Diatomyl® P_{00} (DP_{00}) and Diatomyl® P_2 (DP_2), which were provided by Laffort oenologie. The tested PAP (Rilsan®) were polyamide particles T (PAP_T), polyamide particles MC (PAP_{MC}), and polyamide particles ES (PAP_{ES}), which were provided by Arkema.

Characterization of the particles consisted of water permeability and specific surface area measurements. A permeameter (CECA) was used to evaluate water permeability of the precoat layer, expressed in Darcy unit (A darcy is defined as 1 ml/(sec.cm^2) of a liquid with 1 cp viscosity (water) passing through a 1 cm thick cake at a 1 atm pressure diffential). The specific surface area of particles in m^2/g was measured using BET apparatus (Coultronics).

1. Precoat filtration of enriched wine

A synthetic solution was set in order to test the particles in precoat filtration. This solution could be comparable to the rough wine, which is usually clarified in filter presses using diatomites. The first precoat filtration tests (without body-feed) of enriched white wine were set at 1.10^5 Pa to confirm organoleptic compatibility. The pilot was composed of a column with a diameter of 17 cm and 166 cm in height connected to two tanks of 600 L (figure 1). White wine was used in this study and the variety was Semillon. The white wine used was enriched by adding mannoproteins and aromas (table 1) to facilitate the detection of these compounds. Yeast preparation was added to increase turbidity. The turbidity of the test solution was intentionally high to accentuate the precoat layer plugging. A wine before precoat filtration had a turbidity of approximately 30 NTU while, after clarification, the turbidity was 10 NTU and, in the bottle, the turbidity was less than 1 NTU. The precoat layer, for DP_{00}, DP_2, PAP_T and PAP_{MC}, was composed of 4 kg/m^2 of filtering area, as the industrial operating conditions. For PAP_{ES}, 12 kg/m^2 was used because the precoat layer was not uniform with 4 kg/m^2. For each run, 100 L of enriched wine were filtered.

a) Physical properties

Filtration efficiency was estimated by measurement of both, turbidity (Turbidimeter HACH) and fouling index of the filtrate. The turbidimetric method determines the total suspended solids content of the suspension. The turbidity was expressed in Nephelometric Turbidity Unit (NTU). In the fouling index test, suspension is passed through a standard filter and the rate of blockage is measured. The Fouling Index (FI) was measured

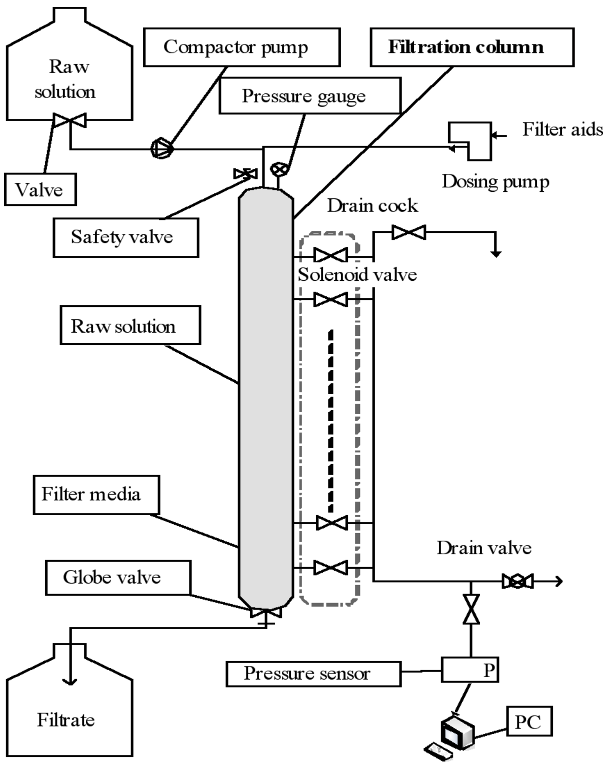

Figure 1 - Pilot plant for filtration experiments

Table 1 - Enriched white wine composition (test 1)

Yeast preparation		1200 ml for 1300 L
Mannoproteins		150 g
Aromas	geraniol	125 mg
	linalol	62 mg
	α-terpineol	125 mg
	citronellol	32,5 mg

according to the method of Descout *et al.* (1976) and was calculated using the equation:

$$FI = T_{400} - 2T_{200}$$

where T_{200} and T_{400} were the time to filter 200 ml, then 400 ml of a sample, respectively at 2.10^5 Pa, on a Millipore membrane (diameter of 2.5 cm, area of 3.9 cm^2 and nominal cut-size of 0.65 µm), in a continuous measurement. If T_{400} exceeded five minutes, the FI was not calculable and the volume filtered in five minutes (V_5) was used. The greater the rate of blockage was, the greater was the amount of colloidal contamination and the greater was the FI (or the smaller is the V_5).

b) Chemical analyses

The aromas and wine molecule concentrations in the filtrate were measured by gas chromatography / mass spectrometry (GC/MS). For the aromas, three extractions were carried out on three different samples. The wine, before and after filtration, was analysed for free sulphur dioxide (free SO_2), titratable acidity (TA), volatile acidity (VA), alcohol and pH. The free SO_2 was determined by the method of Ripper (OIV, 2007, volume 2). The TA was determined by titrating a 5 ml sample free of CO_2 added to dibromo-3',3-thymolsulfonephtaleine (BBT) with 0.1 *N* NaOH and was expressed as g tartaric acid/L. During VA measurement, CO_2 was first removed from a 15 ml sample, the sample was then distilled (Chenard SPINOSA system) and titrated using the Duclaux-Gayon method. VA was expressed as g acetic acid/L (OIV, 2007, volume 1). An ebulliometer (BARUS – SEPCA) was used to determine the alcohol content by volume. Wine pH was measured with a pH-meter (pH 538 Multical® WTW) standardized to pH 7.0 and 4.0. The chromatic characteristics of filtered white wine were analyzed with regard to hue. Therefore, absorbency was measured at 420 nm using an UV/VIS spectrophotometer (V-530 JASCO).

Each experiment and each analysis were repeated at least three times.

Table 2 - Synthetic solution composition (test 2)

Bentonite	0.1%
Arabic gum	0.5%
Leaven	1%
Turbidity	57 NTU
FI (Fouling index)	14 ml in 5 minutes
Solid matter	0.04%

2. PAP regeneration

The second precoat filtration tests were carried out with body-feed at 1.105 in the same filtration column as in 2.1. The aim of these tests was to determine the regeneration of the filter aids using a synthetic clogging solution (table 2) comparable to the rough wine usually clarified using diatomites. The PAP concentration for precoat layer deposit and for body-feed was 1 kg/m^2 and 1 g/L, respectively. Turbidity and FI measurements were used to verify filtration efficiency.

The regeneration procedure consisted of steeping the filter cake in 1% alkaline solution (NaOH) for 12 h followed by three rinses with water.

RESULTS AND DISCUSSION

1. Particle characterization

Filter aid technology represents a compromise between the permeability of the particles deposited and their turbidity removal efficiency (Sulpizio, 1999). Water permeability measurement may help to compare filter aids; when the permeability is low, the filtration is fine. For exemple, Diatomyl P00, which has a permeability of 4.5 Darcy, is usually used for a coarse filtration of rough wine, whereas Diatomyl P2 is employed for a fine filtration because of its permeability of 1.3. When the permeability is about 2 Darcy, the diatomites are used for clarification; and, when the permeability is inferior to 0.5 Darcy, the diatomites are employed for pre-bottling filtration or sterile filtration. Diatomite water permeability measured in this study (table 3) was similar to that obtained by Mota *et al.* (2006) and Tiller (2004). In comparison with diatomite permeability, the new PAP can be classified at different levels (Boittelle *et al.*, 2004) and thus could be used in different filtration stages: PAP_T for clarification and coarse filtration, PAP_{MC} for fine filtration and PAP_{ES} for finer filtration, even pre-bottling filtration. However, the application of PAP in the various stages of winemaking is only possible if the new filter aids are used in adapted industrial equipment.

The results obtained for specific surface area of the filter aids (table 3) confirmed the permeability measurements. The important specific surface areas of diatomites are due to their high internal porosity.

2. Precoat filtration of enriched wine

The analysis of filtration velocity measurements (table 4) shows: i) that PAP_T correspond to coarse filter aids (filtration velocity was higher than that measured with DP_{00}) and ii) that PAP_{ES} filtration velocity was comparable with those obtained with DP_2. Thus PAP_{ES} could be considered as a fine filter aid. However, the quantities of particles used to form the precoat layer were three times greater.

The turbidity measurements (table 5) indicate the presence of suspended particles in the filtrate. All filter aids tested reduced turbidity by at least a factor of 50 % and enhanced filtration efficiency. Turbidity values and FI confirmed the efficiency of fine particles (PAP_{ES} and PAP_{MC}). After filtration on precoat layers formed by D_{P2} or PAP_{ES}, the filtrate FI was measurable. The results obtained with PAP_T show that these can be used as coarse filter aids. Their low turbidity efficiency could be explained by the large pores precoat layer, so small particles could pass through the filter cake.

To determine if the new filter aids modified wine properties after filtration, chemical and usual oenological analyses were performed (table 6). These analyses show that filtration with the new filter aids had no effect on the

Table 3 - Characteristics of tested particles

Particles	Nature	Size (µm)	Specific surface area (m²/g)	Water permeability (Darcy)
D_{P2}	Silica powder	10	1.24	1.3
D_{P00}		15	0.85	4.46
PAP_{ES}	Polyamide powder	< 63	0.55	0.81
PAP_{MC}		$20 < PAP_{MC} < 100$	0.43	3.5
PAP_T		$40 < PAP_T < 200$	0.16	9.27

Table 4 - Filtration velocity obtained during white wine precoat filtration (without body-feed)

	Filter aid	Filtration velocity ($m^3.h^{-1}.m^{-2}$)
Coarse	PAP_T	40.3
	D_{P00}	14.6
	PAP_{MC}	5.5
	PAP_{ES}	2.6
Fine	D_{P2}	2.5

chemical composition of wine (volatile and total acidity, alcohol, pH and free SO_2). The impact of filtration on the wine hue was also observed by measuring OD_{420} (yellow colour intensity). The wine hue decreased according to the degree of filtration (applications), OD_{420} value was reduced with finer filtration.

Since proteins and polysaccharides influence the wine quality and play an important role in its stability, the impact of filtration on the retention of these compounds was evaluated (table 7). Three fractions were selected with regard to molecular weight:

- F1 - molecular weight > 70,000 Da, representing polysaccharides,

- F2 - molecular weight ≈ 40,000 Da, representing mannoproteins,≠

- F3 - molecular weight ≈ 30,000 Da, representing wine proteins.

Polysaccharides were retained similarly by PAP and diatomites, retention was between 13% and 37% (table 7). The behaviour of PAP is surprising and requires further research as the finer particles (PAP_{ES}) retained less than the coarser particles (PAP_T). The phenomenon of adsorption probably occurs in wine protein retention. Mannoproteins were equally retained by PAP and fine diatomites DP_2.

The perception threshold is the minimal concentration, from which, during a triangular test, 50% of tasters recognize the presence of an odorous substance, without being able to identify aroma (Ribéreau-Gayon *et al.*, 2004). The aromas (linalol, α-terpineol, citronellol and

Table 5 - Precoat filtration impact on physical parameters of white wine

	Turbidity (NTU)	*Efficiency* (%)*	FI	V_5 (ml)*	*Efficiency** (%)*
Control	215 ± 5.1	-	-	39	-
D_{P00}	77.4 ± 4.5	*64*	-	75	*48*
D_{P2}	2.4 ± 0.4	*99*	15	-	*90*
PAP_T	149 ± 3.6	*31*	-	45	*13*
PAP_{MC}	21.6 ± 1.2	*90*	-	120	*67.5*
PAP_{ES}	6.45 ± 0.2	*97*	4.5	-	*80*

* V5 represents the volume obtained after 5 minutes of filtration.
** efficiency is calculated as the ratio between the control and the sample measure

Table 6 - Precoat filtration impact on chemical parameters of white wine

	OD_{420}	pH	Free SO_2 (mg/L)	TA (g/L tartaric acid)	VA (g/L acetic acid)	Alcohol content (%)
Control	0.7445	3.7	10	5.8	0.6	11.8
D_{P00}	0.4185	3.7	10	5.8	0.6	11.8
D_{P2}	0.1142	3.7	10	5.7	0.57	11.7
PAP_T	0.6141	3.8	10	5.7	0.54	11.8
PAP_{MC}	0.246	3.8	10	5.7	0.56	11.8
PAP_{ES}	0.2136	3.7	10	5.8	0.57	11.8

Table 7 - Polysaccharide, mannoprotein and protein dosing in white wine after precoat filtration

	Reduction of polysaccharides (%)	Reduction of mannoproteins (%)	Reduction of wine proteins (%)
D_{P00}	*-14.6*	*-11.4*	*-12.0*
D_{P2}	*-30.6*	*-19.8*	*-21.4*
PAP_T	*-36.2*	*-24.9*	*-37.6*
PAP_{MC}	*-36.9*	*-21.2*	*-31.7*
PAP_{ES}	*-13.0*	*-26.9*	*-24.3*

Table 8 - Monoterpene characteristics (Ribéreau-Gayon *et al.*, 2004)

Monoterpenes	Olfactive descriptor	Perception threshold (µg/L)*
Linalol	Rose	50
α-terpineol	Lily of the valley	400
Citronellol	Lemon	18
Geraniol	Rose	130

* Perception thresholds were determined in wine.

geraniol), introduced into the white wine, belong to the terpene family. These compounds are responsible for varietal aroma, due to the grapes, and Muscat-like wines (table 8). The terpenol concentration in simple flavoured wines (Sauvignon, Syrah, Cabernet, Merlot, etc.) is generally lower than the perception threshold. It is necessary to preserve these aromas even during physical treatments like filtration. The filtrate aromatic profile was dependent on the impact of precoat filtration on the retention of these aromas. Table 9 shows the aroma concentrations after filtration with the various precoat layers. The aromatic profiles were then analyzed for each filtrate. The differences were in general not significant.

A panel of tasters also performed the sensorial analysis of wines and no difference could be noticed between each filtered wine.

During similar tests, carried out by Serrano and Paetzold (1994), filtration using diatomites had little effect on the terpenol content. Only the finest diatomite decreased the geraniol content by 10%. This phenomenon was also observed in this study (table 9). After DP_2 precoat filtration, all aromas were decreased (- 8% for linalol, -13% for l'α-terpineol, -18% for citronellol and -13% for geraniol). This could be due to the nature of the fine DP_2 filter aid. The same phenomenon was noted following filtration on PAP_{MC}, where a reduction of 11% for α-terpineol and 14% for citronellol was observed. It should be noted that no significant reduction in aromas was observed during filtration with PAP_{ES}, which is considered to be a fine filter aid.

In conclusion, PAPMC can be used for clarifying filtrations also carried out by DP_2. PAP_T have filtration characteristics comparable to those of DP_{00} and could thus be used to carry out coarse filtrations. However, it should be noted that PAP_T allowed faster filtration than DP_{00}. PAP_{ES} may be employed as a fine filter aid. In addition, tasting results (Boittelle *et al.*, 2005) showed that the polyamide particles did not affect wine taste.

3. Regeneration tests

PAP have the advantage of being available in various grades, they are also inert and are effective as filter aids. In addition, these particles, from their polymeric nature and resistance to alkaline treatment, seem to be regenerable. This was verified during the precoat filtration tests with body-feed of synthetic suspension at 1.10^5 Pa, and was presented in table 10. In the first run, PAP_T were used to form the precoat layer and a mixture of PAP_T-PAP_{ES} was used for the body-feed. The filter cake obtained after filtration was regenerated by alkaline treatment (NaOH) followed by rinsing with water. The regenerated particles (PAPT-PAP_{ES}) were used for the body-feed in the second run. The same operation was repeated twice. The precoat layers in the first and second run were made up of « clean » PAP_T. In the third run, it was made up of a mixture of clean PAP_T-PAP_{ES}.

Logically, the turbidity, FI and filtration velocity values obtained (table 10) were different to those of the preceding tests due to the body-feed and a more clogging suspension. The flux through the regenerated PAP media (run 2) was 50% less than the flux through the clean PAP (run 1), whereas the filtrates were of the same quality. In the last run (run 3), the flux through the PAP_{T+ES} precoat layer was about 60% less than the flux through the PAP_T precoat layer with the same body-feed. This phenomenon, known as « glaçage » (gelling) of the precoat layer, occurs when the precoat layer is less permeable than the cake made of the body-feed. Moreover, the regenerated mix (PAP_{T+ES}) still contained some impurities (such as gelatine), which are not degraded by alkaline treatment. With regard to turbidity and FI values (runs 2 and 3), the filtration was found to be efficient. However, after each run, the filter cake remained partially clogged and the regenerative method could be improved with different cleaning conditions such as various temperatures and the addition of enzymes.

CONCLUSIONS

PAP can replace diatomites as filter aids in wine filtration as they were found to be effective (reduced turbidity and enhanced FI). These tests also showed that PAP do not affect the chemical parameters of the wine during filtration. PAP_T can be compared with DP_{00} regarding filtration efficiency, filtration velocity was higher with these filter aids. PAP_{ES} and PAP_{MC} were considered to be finer filter aids. Organoleptic qualities of the wines were little affected by filtration using PAP. PAP also have the advantage, compared to diatomites,

Table 9 - Aroma dosing of white wine filtrate obtained after precoat filtration (µg/L)

	Linalol	α-terpineol	Citronellol	Geraniol
Control	211 ± 19	415 ± 7	108 ± 4	430 ± 18
D_{P00}	204 ± 4	401 ± 4	103 ± 1	412 ± 17
D_{P2}	192 ± 9	342 ± 52	79 ± 21	389 ± 44
PAP_T	215 ± 3	458 ± 8	105 ± 11	449 ± 12
PAP_{MC}	205 ± 2	369 ± 14	93 ± 5	447 ± 43
PAP_{ES}	207 ± 18	399 ± 12	99,82 ± 2	401 ± 9

* It should be noted that the aromas are added in quantity higher than the perception threshold.

Table 10 - Regeneration of polyamide particles (P = 1.10^5 Pa., S = 0.024 m^2)

Run	Precoat (2 kg/m^2)	Body-feed (100 g/hL)	Total filtered volume (L)	Turbidity reduction (%)	FI improvement (%)	Filtration velocity (m^3/h.m^2)
1	PAP_T	PAP_T-PAP_{ES}	550	96	> 87	4
2	PAP_T	Regenerated PAP_T-PAP_{ES} (alkaline treatment)	40	98	> 80	1.5
3	PAP_T-PAP_{ES}	Regenerated PAP_T-PAP_{ES} (alkaline treatment)	50	86	> 73	0.9

of requiring no preliminary rinsing before use (Boittelle *et al.*, 2005). The tested particles could be used for the same filtration processes as diatomites.

This study highlights several advantages of these new PAP filter aids. They are of vegetal origin, safe for end-users, can be re-used several times and are up-graded by incineration without volatile organic carbon (VOC) release. The calorific value, which is given in the FDS, is 35 MJ/kg (8,370 kcal/kg). Thus precoat filtration using PAP results in the reduction of both waste quantity and ground contamination. Additional experiments could realized on red wine and on further grape variety to validate our conclusions.

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