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# Alginite – A Novel Filter Aid in Precoat and Cake Filtration

Presently, diatomite is the dominant filter aid in the food and beverage industry due to its high porosity and satisfactory filtration resistance. However, its use is also regarded critically because of carcinogenic dust, possible trace emissions of heavy metals such as arsenic and, as an outcome of intensified environmental regulations, a future increase of disposal costs of the filtration waste. Therefore, improving cake filtration through the application of new filter aids and techniques substituting or reducing diatomite is a dynamic field of research.

The present work was carried out using a new material as filter aid for beer filtration based on the natural rock alginite. Alginite is an immature oil shale from Gércse, Hungary. It consists of fossil organic and inorganic matter. To ensure compliance with the quality requirements and for the purpose of improving filtration properties of alginite, the material has to be thermally activated and washed. The obtained material A1000 is free from organic matter, shows only minor ion leaching and no swellability. In a laboratory filter system, beer was filtered with A1000 and diatomite as reference. The A1000 as filter aid leads to short filter service life because of a higher cake resistance due to  $\beta$ -glucan retention. Cake resistance was successfully reduced by adding commercial filter aids, such as diatomite and perlite, respectively. The smallest cake resistance was observed upon mixing A1000 and perlite. A bright and stable beer was obtained using both A1000 and A1000 mixtures, what brings industrial filtrations with long filter service life within reach. Even more important, A1000 removes polyphenols during filtration, which is beneficial for an enhanced beer stability.

From the disposal point of view, the agricultural use of filter cakes appears promising, because both alginite and perlite are well-proven soil conditioners. Hence, alginite use has the prospective of realising filtration processes in food and beverage industry with enhanced economy.

Descriptors: beer filtration, filter aid, cake filtration,  $\beta$ -glucan, polyphenol

## 1 Introduction

In the food and beverage industry, cake filtration is state of the art to obtain a clear and stable product, e.g. in beer, wine and juice production. In order to realise a long filter service life with high flow rates a filter aid is used. At present, diatomite, fossilised remainders of diatoms, is the dominant filter aid due to its high porosity.[1] The disadvantages of diatomite are: carcinogenic dust [2], trace emissions of heavy metals [3, 4] and rising disposal costs of the filter cake, because of intensified environmental regulations.[5, 6] Some improvements concerning these problems were achieved by special pre-treatment of the diatomite, which however are regarded insufficient so far. Therefore, optimisation of cake filtration by means of the use of other filter aids and techniques without diatomite is an important field of research. Particularly in the last few years the focus of investigations has been on cellulose, cellulose mixtures and their recyclability.[7–9] Also, new synthetic materials made of polystyrene and polyvinyl polypyrrolidone (PVPP) which combine

filtration supporting and stabilising properties were developed. [10] These substances find widespread application in the Asian region, but not in Europe. Here, classical filter aid alternatives such as cellulose, perlite and clays are used. However, all mentioned materials are either expensive, need a different process design or do not provide a filtration performance like diatomite.

The present work was carried out using a new filter aid for beer filtration based on natural rock alginite. Alginite (sensu Jámor & Solti, 1975), an immature oil shale from Hungary (Gércse) with an organic content of up to 19 % results from fossil yellow-green algae of the genus *Botryococcus braunii*. It is mined in open pits as raw material with a capacity of 1 mill t/a. At present, it is used mainly in agriculture as a soil improving agent.[11–13] Therefore, an agricultural use of the filtration waste is intended which would enhance beer filtration economy by more advantageous disposal. In order to investigate whether alginite might constitute a suitable alternative for commercial filter aids in cake filtration, characterisation and filtration experiments with alginite, pre-treated alginite, diatomite, perlite and mixtures of them were carried out. Furthermore, it is reported that immature oil shales such as alginite can remove organic substances from aqueous solutions.[14, 15] Therefore the interaction with mixed linkage (1-3,1-4)- $\beta$ -D-glucans, commonly known as  $\beta$ -glucans and polyphenols has been investigated.  $\beta$ -Glucans are well known to have an adverse effect on beer filterability [16, 17] while polyphenols, impair beer stability

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due to the formation of protein-polyphenol complexes.[18, 19] The quality requirements for filter aids, specific cake resistance, reduction of  $\beta$ -glucan and polyphenols, respectively, were used to evaluate filtration performance.

## 2 Materials and Methods

### Materials

Alginite was provided by Terra Natural Resources GmbH, Germany, in a grain size of 3 mm. Commercial filter aids diatomite (Becogur 200 and Becogur 3500) and perlite (Becolite 3000) were derived from E. Begerow GmbH, Germany. The unfiltered beer was supplied by Freiberger Brauhaus GmbH, Germany.  $\beta$ -glucan analysis was done with the  $\beta$ -glucan assay kit (Mixed Linkage) from Megazym, Ireland. Hydrochloric acid p.A. was obtained from VWR, USA, and PES-filter by Macherney-Nagel GmbH & Co. KG, Germany.

### Equipment

Laboratory scale filtration experiments were carried out in a filter plant with a filter area of 1.23 dm<sup>2</sup> (1), as shown in figure 1. It was equipped with three pressure transmitter DT1 (9) by Titec GmbH, Germany, and a FCH-m-PVDF-HD flowmeter (10) by Biotech, Germany. As pumps BVPZ (7) by Ismatec, Germany, and dosing

pump (8) by ProMinent, Germany, for beer and filter aid dosage were used, respectively. The unfiltered beer was cooled using a cooler (2) by Cornelius Deutschland GmbH, Germany. Thickness and humidity of the filter cake were measured by a digital calliper Precise PS 7215 by Burg Wächter, Germany, and a halogen balance HB43-S by Mettler Toledo, USA, respectively. Particle size distribution was analysed by static light scattering measurement conducted on a LS 13320 laser diffraction particle size analyser by Beckman Coulter, USA. Sieving analyses were carried out with a sieve shaker AS100 and a 100 $\mu$ m sieve from Retsch GmbH, Germany. ICP measurements were carried out using an iCAP 6500 Duo ICP-OES by Thermo Fisher Scientific GmbH, Germany. Thermogravimetric analyser TGA/DSC 1 with a mass-flow controller GC 200 by Mettler Toledo, USA, was coupled to a quadrupole mass spectrometer ThermoStar<sup>TM</sup> by Pfeiffer Vacuum, Germany. UV/Vis-analysis was performed using a 100-QS cuvette by Hellma Analytics, Germany, and a V-630 spectrometer by Jasco, Germany, and SEM-EDX analysis was done by using a JEOL JSM-7001F, Japan.

### Alginite pre-treatment

The material was calcined at 1,000 °C for 2 hrs and washed afterwards with a 0.1 M HCl solution to remove leachable metals. The obtained material is called A1000 and was then ground into less than 100  $\mu$ m small particles after drying at 105 °C.

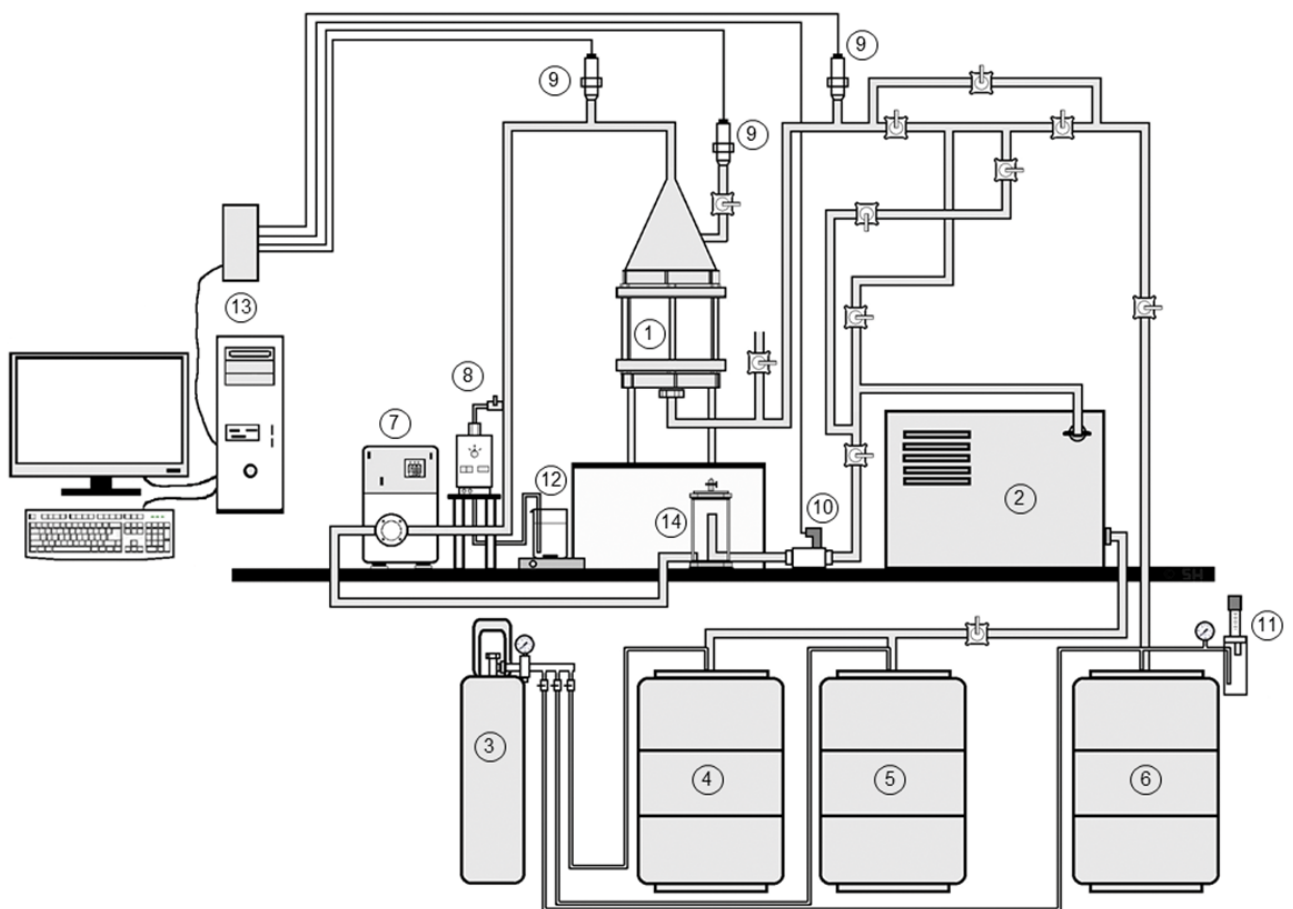


Fig. 1 Laboratory scale filtration plant; Filter (1), cooler (2), carbon dioxide (3), KEG with water (4), KEG with unfiltered beer (5), KEG for filtrate (6), pump (7), dosing pump (8), pressure transmitter (9), flowmeter (10), bung device (11), dosage mixture (12), data acquisition (13), vent (14)

### Determination of particle size

The optical model Fraunhofer.rf780d with PIDS Data was used for the evaluation. The measuring concentration was 7–8 % (42–44 PIDS), measurement time 60 sec and pump speed 45 %. Every sample was measured in water and treated for 60 sec with ultrasound beforehand.

### Determination of ion leaching

For analysis of beer-soluble elements 1 g filter aid was suspended in 10 ml beer and shaken overhead for 24 hrs. Beyond that time period solid matter was removed from the beer suspension by a 0.2 µm PES-filter. Metal concentrations in the filtrate were determined by ICP-OES.

### Determination of ignition loss

About 20 mg substance were placed in a 70 µl alumina crucible with perforated lid and heated from 25 °C up to 1,000 °C in a TGA device with a heating rate of 10 K/min in a pure nitrogen flow of 50 ml/min. Difference between initial and end weight at 1,000 °C yields the ignition loss of the material.

### Filtration and determination of specific filter cake resistance and porosity

Filtration experiments were carried out in duplicate under constant flow conditions (7.5 hl/(h·m<sup>2</sup>)) in a laboratory plant with a fine metal tissue as filter means. Flow resistance of the tissue is negligible compared to that of the filter cake. For the first precoat (2 kg/m<sup>2</sup>) coarse diatomite (Becogur 3500) or perlite (Becolite 3000) was added to act as support layer for the filter cake. This aggradation was performed by pumping water in a closed circuit through the plant. The compositions of the filter aids during the different experiments are listed in table 1.

**Table 1** Cake composition of filtration experiments

Experiment	Support layer (precoat)	Composition of dosage		
		Diatomite	A1000	Perlite
1	Becogur 3500	100 wt.-%	–	–
2	Becogur 3500	80 wt.-%	20 wt.-%	–
3	Becogur 3500	60 wt.-%	40 wt.-%	–
4	Becogur 3500	40 wt.-%	60 wt.-%	–
5	Becogur 3500	20 wt.-%	80 wt.-%	–
6	Becogur 3500	–	100 wt.-%	–
7	Becolite 3000	–	80 wt.-%	20 wt.-%
8	Becolite 3000	–	60 wt.-%	40 wt.-%
9	Becolite 3000	–	40 wt.-%	60 wt.-%
10	Becolite 3000	–	20 wt.-%	80 wt.-%
11	Becolite 3000	–	–	100 wt.-%

When deposition of the precoat was complete, the liquid was switched from water to unfiltered beer. Based on filtrate turbidity and pressure increase within the filter, filter aid dosage or filter

aid mixtures were adjusted. The pressure sensors were placed upstream, within and downstream the filtration chamber to determine pressure drop across the filter. Filtrate pressure was kept at 1.4 bar to prevent gas release. After filtration was complete, thickness (L) of the filter cake was measured to calculate the volume fraction of the cake ( $\phi_{k,f}$ ) during the process. During the experiments the following equation (Eq. 1 acc. to [20]) was used for the calculation of specific cake resistance ( $r_L$ ) because of slight pressure fluctuations.

$$\Delta p \cdot A \cdot \frac{dt}{dV_f} = \frac{r_L \eta_l \phi_{k,f}}{A} \cdot V_f + R \eta_l = m \cdot V_f + b \quad (\text{Eq. 1})$$

$$\text{with: } \phi_{k,f} = \frac{A \cdot L}{V_f} \quad r_L = \frac{m \cdot A}{\eta_l \cdot \phi_{k,f}} \quad R = \frac{b}{\eta_l}$$

The specific filter cake resistance ( $r_L$ ) and filter medium resistance (R) can be determined from the slope (m) and the ordinate intercept (b) of filtration equation diagram, respectively. [20]

Average cake porosity of the filter cake was calculated by equation 2 [20]. For this purpose, the wet to dry cake mass ratio  $m'$  can be determined by weighing the cake first, dewatering the cake by drying and weighing the dried cake.

$$\varepsilon = 1 - \left[ \frac{\rho_s}{\rho} (m' - 1) + 1 \right]^{-1} \quad (\text{Eq. 2})$$

### Analysis of bitter units, β-glucan and polyphenols

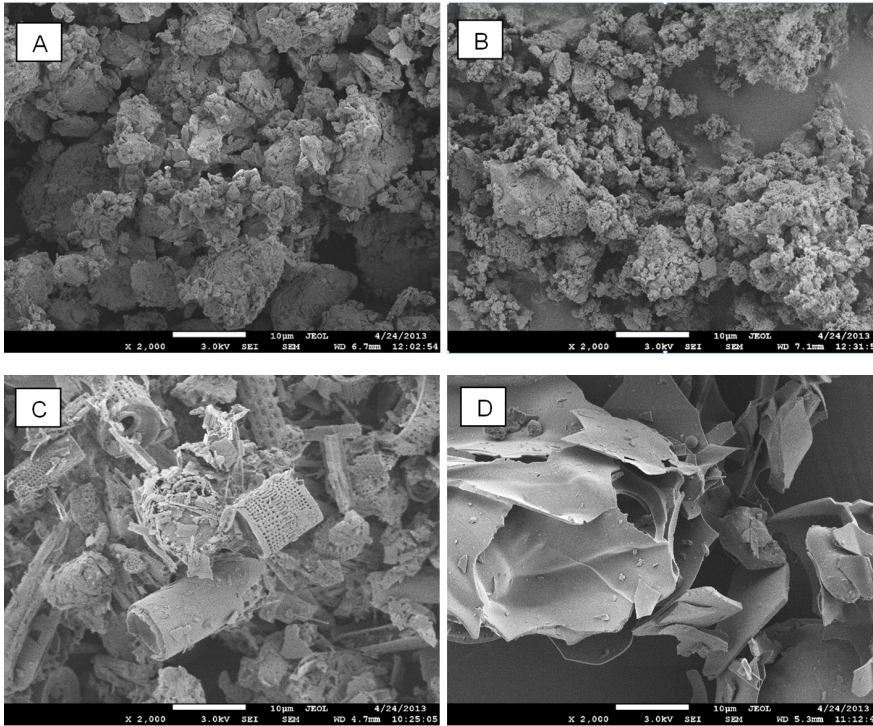
Bitter units analyses were carried out according to MEBAK II 2.18.1 (EBC-method).[21] β-Glucan content in beer filtrate was determined with the help of an enzyme kit according to EBC Method 8.11.1. [22] For total polyphenol content analysis different amounts of A1000 were suspended in diluted gallic acid (236 mg/l; pH 4.5) and shaken for 4 hrs. After centrifugation of the suspension for 10 min at 1,200 rpm the supernatant was removed and analysed according to MEBAK II 2.17.1.[21]

## 3 Results and Discussion

### Characterisation of alginite and A1000

Alginite, a crude oil shale, consists of inorganic and organic components. In order to verify, whether the material can be used as filter aid, ion leaching, particle sizes, surface area and ignition loss were examined. Table 2 summarises the results for alginite, calcined alginite, diatomite and perlite.

Due to the high organic content, up to 19 wt.-%, and carbonates, up to 20 wt.-%, the dry material exhibits an ignition loss of 24 wt.-%. [11] This and the high ion leaching of the material, especially for aluminium, iron and calcium, are in contradiction with the quality requirements for filter aids.[23] Therefore a pre-treatment of alginite is necessary. To minimise these critical parameters alginite was calcined at 1,000 °C and washed with hydrochloric acid (0.1 M). In table 2 alginite and calcined alginite (A1000) are compared with commercial qualities of diatomite and perlite and the quality requirements for diatomite according to Gock et al.[23]



**Fig. 2** SEM-images of alginite (A), A1000 (B), diatomite Becogur 200 (C) and perlite Becolite 3000 (D)

As shown in table 2, A1000 fulfils the quality requirements for filter aids. Leaching of beer-soluble elements and the ignition loss have been reduced successfully by the pre-treatment step. Particularly the iron, aluminium and calcium concentration decreased significantly. Nevertheless, even though the calcium and magnesium concentration lies under the limit of the quality requirements, these elements might be critical when the pre-treatment is incomplete, due to their haze and taste influencing properties.[24, 25] It has to be kept in mind that the contact time of the substances with the beer during leaching tests was 24 hrs, a duration which usually occurs not in industrial filtrations. During laboratory filtration tests the dosage of A1000/diatomite mixtures was 150–250 g/hl while for A1000/perlite mixtures a dosage of 150–300 g/hl was used. Hence, ion leaching A1000 has a minor influence on product quality. For all tested substances the concentration of arsenic, an element which stands in the focus of public perception [4], was under the detection limit (< 50 µg/l). Moreover, this process reduced the specific surface

of alginite. As depicted in figure 2 the surface sintered and the particles sizes decreased. Only small holes are visible where the organic matter and the carbonates had been located before. A porous structure similar to diatomite was not received. Smallest surface area was measured for perlite particles (Becolite 3000) which has a shard-like structure. Hence, specific surface area of A1000 lies between perlite and diatomite.

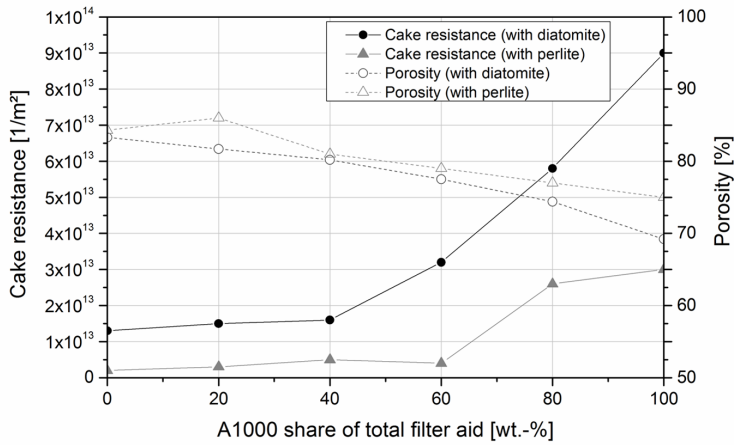
## Filtration Experiments

To investigate the applicability of A1000 as filter aid in precoat and cake filtration, laboratory scale filtration experiments with different filter aid compositions were carried out. When using A1000/diatomite mixtures the dosage was 150–250 g/hl, whereas for A1000/perlite mixtures a slightly higher amount (150–300 g/hl) was used. The amount of dosage increases with increasing A1000 share within a test series. As shown in figure 3 average porosity of the filter cake slightly depends on the A1000 amount in the filter aid mixture. During beer filtration experiments with increasing A1000 amount porosity of diatomite

mixtures decreased by 17 %, while with perlite a decrease of 11 % was determined. The specific cake resistance for A1000/diatomite mixtures slightly increased up to 40 wt.-% A1000. At higher concentrations, the resistance increased fast from  $1.5 \cdot 10^{13}$   $1/m^2$  to  $9.0 \cdot 10^{13}$   $1/m^2$  for pure A1000. The higher the cake resistance the shorter the filter service life. Cake resistance values over  $5.0 \cdot 10^{13}$   $1/m^2$  lead to short filtration periods due to the fast increasing pressure. Therefore, the use of A1000/diatomite mixtures is advisable as filter aids in filtration processes only for low quantities of A1000. The cake resistance for A1000/perlite mixtures increased more slowly. Up to 60 wt.-% A1000 the resistance was approximately  $0.5 \cdot 10^{13}$   $1/m^2$ . Even with pure dosage of A1000 and a support layer from perlite the resistance did not exceed  $3.0 \cdot 10^{13}$   $1/m^2$  during the beer filtration. The difference in cake resistance of the two 100 wt.-% A1000 experiments come from the different pre-coats. Therefore, A1000/perlite mixtures up to 60 wt.-% A1000 make a longer filter service life with high flow rates possible.

**Table 2** Comparison of fine alginite, A1000 with commercial filter aids Becogur 200 (diatomite) and Becolite 3000 (perlite) and the quality requirements for diatomite according to Gock et al. [23]

	Alginite	A1000	Becogur 200 (Diatomite)	Becolite 3000 (Perlite)	Quality Requirements acc. to [23]
<b>Beer-soluble Metals [mg/ 100g]</b>					
Al	15	5	7	5	7.5
As	< 0.05	< 0.05	< 0.05	< 0.05	0.5
Ca	102.2	48.5	19.0	2.4	50
Fe	15	0.4	1.8	2.0	5
Mg	42	35	4.6	1.1	–
Na	6.3	4.2	14	22	–
<b>Specific surface area [m<sup>2</sup>/ g]</b>	34.0	7.8	13.5	2.2	–
<b>Mean particle size, d<sub>50</sub> [µm]</b>	12.9	7.3	17.9	42.8	12–25
<b>Ignition loss</b>	24 wt.-%	< 1 wt.-%	< 1 wt.-%	< 1 wt.-%	< 2 wt.-%

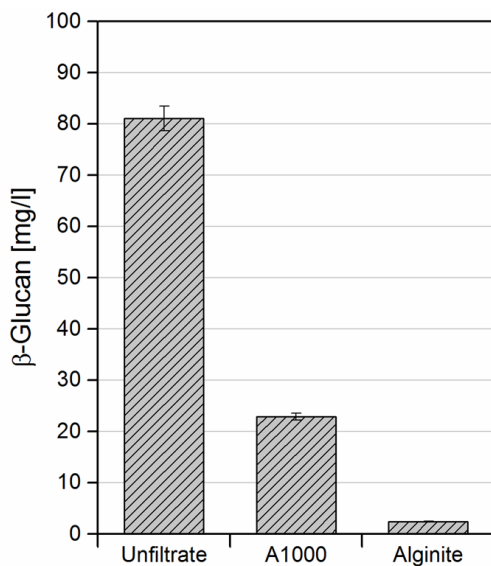


**Fig. 3** Dependence of the porosity and the specific cake resistance from A1000 share in the dosage; rest of the dosage is diatomite (Becogur 200) and perlite (Becolite 3000), respectively

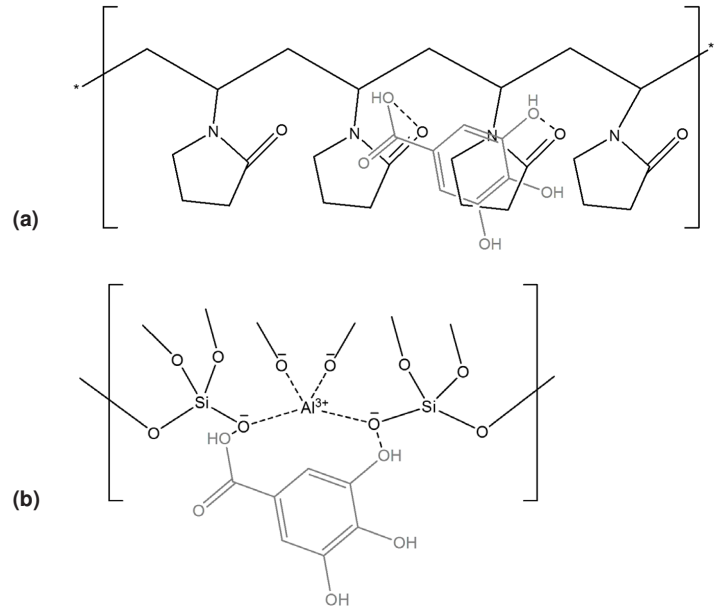
**Investigation of  $\beta$ -Glucan and Polyphenol interaction with A1000**

It was found out that calcined alginite is able to reduce  $\beta$ -glucans. This substance class can complicate the filtration process by gel formation through blocking pores of the filter cake. [25] This causes pressure to increase fast and results in a short filter service life. As depicted in figure 4,  $\beta$ -glucan concentration can be reduced by 70 % using 20 wt.-% pure A1000 without any other filter aid. When the share of A1000 decreases, less  $\beta$ -glucan was removed. The retention of this substance class affects the filtration process adversely and leads to higher cake resistance of filter aids with high A1000 portion. It is for this reason that during filtration experiments the amount of the dosage had to be increased with increasing A1000 share of total filter aid to allow longer filter service life with high flow rates. The exact mechanism of the adsorption of  $\beta$ -glucan by Alginite and A1000, respectively, is not fully understood, yet.

Beer filtration has not only the purpose of achieving a bright product; it also serves to enhance product stability. Therefore, during beer



**Fig. 4**  $\beta$ -Glucan reduction using 20 wt.-% pure filter aid A1000 and untreated alginite

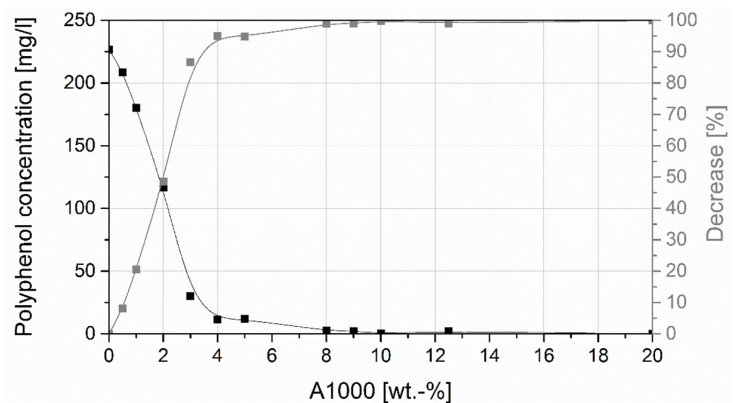


**Fig. 5** Interaction of polyphenol (gallic acid) with PVPP acc. to [18](a) and A1000 (b)

filtration polyphenols, which form complexes with proteins, are removed by standard procedure with PVPP.[18, 26] The interaction between polyphenol and PVPP is illustrated in figure 5a. The adsorption of polyphenols by PVPP is based on hydrogen bonds. The formation of such bonds between the mineral structure of A1000 and the polyphenols is possible, too (Fig. 5b). In order to investigate, whether the novel filter aid can reduce polyphenols, too, adsorption experiments were carried out with gallic acid, which is a standard compound for polyphenols.

As shown in figure 6, calcined alginite A1000 is suitable to completely remove polyphenols from beer, when high amounts of A1000 (10 wt.-%) or more were used. Even 2 wt.-% A1000 reduced polyphenol concentration by half. Hence, A1000 might contribute to beer stability when it is used in the filter aid mixture. Nevertheless, because of the high dilution of A1000 during beer filtration, the reduction of polyphenols is limited. This is why the beer stabilising step with PVPP cannot be waived, but facilitated.

Bitter substances (iso- $\alpha$ -acids and  $\beta$ -acids) from hop might bind to A1000 like polyphenols due to their similar chemical structure. Also, some polyphenols can impart bitterness to beer, too.[18] This



**Fig. 6** Reduction of polyphenols in beer depending on A1000 amount (weight by volume)

**Abbreviations**

A	filter area
A1000	calcined alginite
BU	bitter units
$\varepsilon$	average cake porosity
L	thickness of filter cake
$m'$	wet to dry cake mass ratio
$\eta_l$	viscosity of beer at 0 °C
PVPP	polyvinyl polypyrrolidone
$\Delta p$	pressure difference before and after the filter
$\rho_s$	density of particles (filter aid)
$\rho$	density of filtrate (beer)
$r_L$	specific cake resistance
R	filter medium resistance
$\Phi_{k,f}$	volume fraction of the cake
$V_f$	volume of filtrate

is why the filtrated beer was found to display reduced bitterness. The results of bitter unit (BU) analyses showed that an increasing A1000 share in the dosage leads to a higher loss of bitterness, up to 30 % when using A1000 as sole filter aid. BU decreased from 28.1 for diatomite and perlite, respectively, to 19.8 for A1000. Therefore, to achieve the same taste there are two alternatives: (I) A1000 portion in the filter aid does not exceed 50 % (-13 % BU) or (II) stronger hopping during beer production. However, because it has still not been proven that A1000 reduces all bitter substances equally, it is possible that the ratio between different hop compounds can be changed, in particular  $\alpha$ -acids and hop oils [25]. This would lead to a different taste. Therefore, a control by a taste panel is matter of forthcoming activities.

## 4 Conclusion

Cake filtration with the dominant filter aid diatomite is going to become more expensive due to the intensified disposal regulations of the filtration waste. The novel filter aid A1000, pre-treated alginite, fulfils the requirements of a filter aid. It is not swellable, shows a minor ion-leaching and improves the cake permeability during filtration. While the specific cake resistance of pure A1000 is still too high to provide a long filter service life, the resistance for mixtures with common filter aids is significantly decreased. The filter aid mixtures of calcined alginite (A1000) and perlite showed a lower cake resistance compared to similar mixtures with diatomite, in particular for high proportions of A1000. The used amount of dosage slightly exceeded the range of common filter aids (+ 50 g/hl). Therefore, A1000/ perlite mixtures not only provide a diatomite-free filtration with high flow rates and a long filter service life, but also might improve the stability of the product by removing polyphenols. A bright product was obtained when using both A1000 and A1000 mixtures.

Nevertheless, bitter substances are reduced, too, when the filter aid contains high amounts of alginite. To overcome this problem,

A1000 is proposed to be used in the wort prior to boiling and hop addition. The advantage of this procedure would be a reduction of  $\beta$ -glucans before the filtration, which might increase the filter service life. For a better evaluation of the results and the proposed process further analyses of the filtrated wort and beer, respectively, and a control by a taste panel are matter of follow-up investigations.

However, the agricultural use of the filtration waste seems to be promising because alginite and perlite are already used as soil conditioner. This will lead to decreased disposal cost compared to a disposing of the material in a landfill or else.

Thus, in combination with the high flow rates and long filter service life, the filter aid mixtures based on alginite have the potential of realising filtration processes in food and beverage industry with enhanced economy.

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