HARD SELTZER BASE

Color and odor reduction with activated carbon

NOTC
Product names and filter sheet grades may have changed since the application note was created.

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Content

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Abstract

Commonly, hard seltzer is produced from sugar cane or malt-based alcohol, which is obtained by fermentation. Depending on customer preferences and legal requirements, alternative sources for the alcohol base can be used, for example cider. After the fermentation process, all fermentation-based alcohol fractions exhibit some unwanted solid particles, colorants, and odorants. Since depth filtration is regarded as one of the most efficient and cost-effective filtration methods, FILTROX depth filter sheets are used to remove any remaining and unwanted components from hard seltzer as they influence the flavor of the final product. This application note describes the evaluation of a treatment of cider as a hard seltzer base with immobilized activated carbon on laboratory scale and pilot scale to achieve the optimum desired color and odor reduction of the hard seltzer base. On a laboratory scale, three different immobilized activated carbon products, FILTROX CARBOFIL™ CA, CARBOFIL™ RW and CARBOFIL™ RHC, were used for the activated carbon treatment step to reduce the color of the cider base below 0.5 EBC. Based on the results of the lab scale treatment, 600 L cider were treated with CARBOFIL™ CA depth filter sheets, resulting in a clear and transparent hard seltzer base. Additionally, the filtrate was examined for the removal of various fatty ethyl esters, as some of the major contributors to the typical aroma of cider. A reduction of close to 100 % was achieved for ethyl decanoate (378.2 µg/L) and ethyl dodecanoate (18.4 µg/L), reducing the initial concentration below the limit of quantification (LOQ). The concentration of ethyl hexanoate (- 93%) and ethyl octanoate (- 98%) were significantly reduced from 389.5 µg/L to 28.5 µg/L and from 590.0 µg/L to 13.6 µg/L respectively. A smaller decrease of 12 % from 263.5 µg/L to 233.0 µg/L of ethyl butyrate was measured. Overall, the presented data show the effectiveness of a treatment with immobilized activated carbon for color and flavor removal in the production process of a hard seltzer utilizing cider as an alcohol base.

1 The Challenge

The term 'hard seltzer' is used to describe a type of low-calorie beverage that generally has an alcohol content of around 4-6 % ABV. The hard seltzer beverage sector has experienced exceptionally high growth rates over the last few years and is continuing to gather significant momentum in 2021. One of the key factors to its success is the distinguished and elevated taste experience for the customer. To meet customer expectations for a clear, crisp, and fresh tasting product, the preparation of a hard seltzer base with no color, flavor or aroma is the ultimate goal. Differences in raw materials and difficult fermentation conditions pose a challenge in achieving the highest quality in the final product, as a wide variety of substances are produced during the fermentation process that affect both color and odor. In the US market, fermenting a sugar solution to produce a hard seltzer base is common practice. The impact of a treatment with FILTROX CARBOFIL™ immobilized activated carbon on such a sugar-fermentation hard seltzer base has been presented by Maxminer and Rettberg (2021). Excellent color removal and flavor compound adsorption resulted in a clear and almost neutral hard seltzer base.

With the increasing popularity of hard seltzer, alternative bases are used for production. Utilizing cider as a base proves to be very common, especially in the European market. This might be affected by regulatory objections and tax classifications of hard seltzer bases (Nadolski and Schock 2021). Different fermentation conditions impact the flavor profile of a cider (Villière et. al. 2015) and various esters form one of the major groups of contamination on the way to a clear and crisp hard seltzer base. The cider base usually exhibits wine-like and/or apple-like characteristics, fermentation flavors, slight turbidity, and coloration. In the following application note, the impact of activated carbon treatment of a cider base is described. The goal is to remove the yellowish color and the apple-like odor by using different

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CARBOFIL™ depth filter sheets which contain immobilized activated carbon and therefore combine the high adsorption capacity of activated carbon with the simple and safe handling of depth filter sheets.

2 Materials & Methods

The materials used and the respective methods applied are described in this chapter for both, the laboratory and the pilot scale filtration trials.

2.1 Laboratory scale filtration

2.1.1 Set-up

The test system for laboratory scale filtration was set up with a peristaltic pump (Baoding Shenchen Precision Pump Co. LTD. with pump head YZ1515x), a pressure gauge (TRI-MATRIC AG) and a FILTROX 2" Mini Capsule. The corresponding FILTROX CARBOFIL™ depth filter sheet was inserted into the Mini Capsule. The inlet of the Capsule was connected to the peristaltic pump, with the pressure gauge in between, with a Silicone tubing #17 (Shenchen Precision Pump Co. LTD.). The pressure gauge is necessary as a maximum differential pressure should not be exceeded. Since the filtration with immobilized activated carbon is based on adsorption properties, an increase in pressure is not desired. If a significant pressure increase is measured, a prefiltration step would be necessary. The schematic set-up is shown in figure 1.

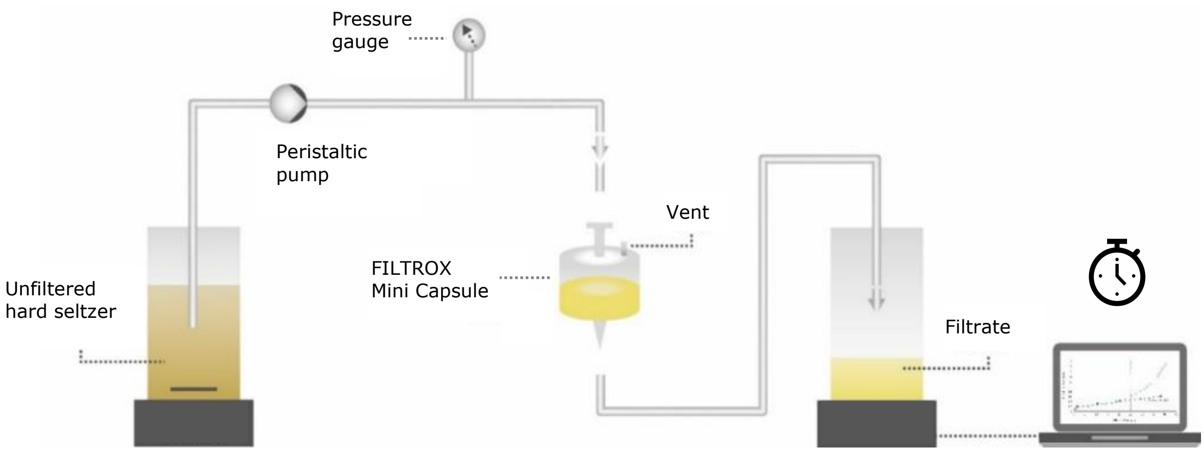

Figure 1: Schematic overview of the test set-up.

2.1.2 Rinsing

To wash out any loose activated carbon particles from the depth filter sheet, the sheets were rinsed with 50 L/m² of water at a flow rate of 300 L/m²×h prior to each filtration test. Since the filtration area on a lab scale is 0.0021 m², this corresponds to a rinsing volume of 100 mL.

2.1.3 Filtration

Depending on the turbidity value of the unfiltered product, a prefiltration prior to the activated carbon treatment step will be necessary to maximize the absorption capacity of the activated carbon sheets. For this purpose, FILTROX offers the standard depth filter FIBRAFIX® AF series. For this trial, the cider was already pre-filtered (see tube on the left in figure 2), thus, no prefiltration was applied.

2.1.4 Activated carbon treatment

As the three filter sheet types CARBOFIL™ RW, RHC and CA have so far proved successful for decolorizing and odor reduction of hard seltzer, these sheets were chosen for the experiments in the laboratory scale. The filter sheet types differ in the types and quantities they contain of immobilized activated carbon and filter aids. Beside the purified cellulose, CARBOFIL™ RW also contains filter aids that enable the removal of a small number of solid particles. However, this sheets also has a lower activated carbon content than that of CARBOFIL™ CA and RHC. Further information about the sheet types can be found in the respective product specifications.

To assess the filtration performance of all filtration experiments, the filtration flux and the differential pressure were recorded over time. The filtration flux is a very important parameter for treatment with activated carbon filter sheets. Since the filtration is based on an adsorption mechanism and the colour and flavor molecules need sufficient contact time with the activated carbon, a maximum filtration flux of 150 L/m²×h is recommended.

2.2 Pilot scale treatment

The subsequent pilot scale test was performed with the CABROFIL™ CA filter sheet. A 600 L batch was filtered according to the instruction manual for filter sheets and a plate and frame filter with filter sheet dimension of 60 × 60 cm was used. After inserting the immobilized activated carbon sheets, the filter was rinsed and sterilized with hot water for 20 min with a constant water temperature of 80 °C at the outlet of the filter. After the sterilization process, the filter was cooled down with cold water and purged with CO_2 to push out the remaining water. Following sterilization and purging, the cider was feed to the filter with a pump at a flowrate of 150 L/m²×h.

2.3 Analytics

To evaluate the performance and find the best suited filter sheet type for this application, various analytics were carried out. For the laboratory scale, the evaluation was based on color removal and for the pilot scale trials, color and flavor removal were evaluated by the below described analytical methods.

2.3.1 Color

The absorption was measured in 1 cm UV/VIS cuvettes at 430 nm with a Hitachi U-1900 spectrophotometer, according to Analytica-EBC, Section 9 Beer, Method 9.6. A double determination was carried out. The EBC value was calculated based on the measured absorption value (see equation 1). An EBC value of ≤0.5 EBC should be achieved after the treatment.

$$EBC = Abs[430\ nm] \times 25 \qquad [1]$$

2.3.2 Flavor analysis

Ethyl butyrate, ethyl hexanoate, ethyl octanoate, ethyl decanoate and ethyl dodecanoate in the filtrate were determined by headspace-solid phase microextraction (HS-SPME) gas chromatography mass spectrometry (GC-MS). Esters were extracted from 2 mL liquid sample using a 50/30 µm DVB/CAR/PDMS fiber, the column used for chromatographic separation was a HP-5MS UI column (30 m × 0.25 mm i.d. × 0.25 µm film thickness). The extraction and GC parameters were used as described by Dennenlöhr et. al. (2020). Isotopically labelled d5-ethyl hexanoate was used as internal standard at a concentration of 100 µg/L.

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3 Results and Discussion

3.1 Laboratory Scale Trials

300 mL were filtered in each trial. A comparison of the unfiltered cider base and the filtrate after CARBOFIL™ CA treatment is shown in figure 2. Over time, no pressure increase was measured during any of the three trials. The EBC values of each 50 mL fraction are shown in figure 3. Compared to the initial color of 3.6 EBC, after the activated carbon treatment all 50 ml fractions had a color of below 0.5 EBC. Overall, the color removal was very similar for all three sheet types. CARBOFIL™ CA showed the best color removal for the first 5 fractions. The target values of ≤0.5 EBC was achieved in all fractions for all three products.

Figure 2: Unfiltered vs. filtered (CARBOFIL™ CA) cider base.

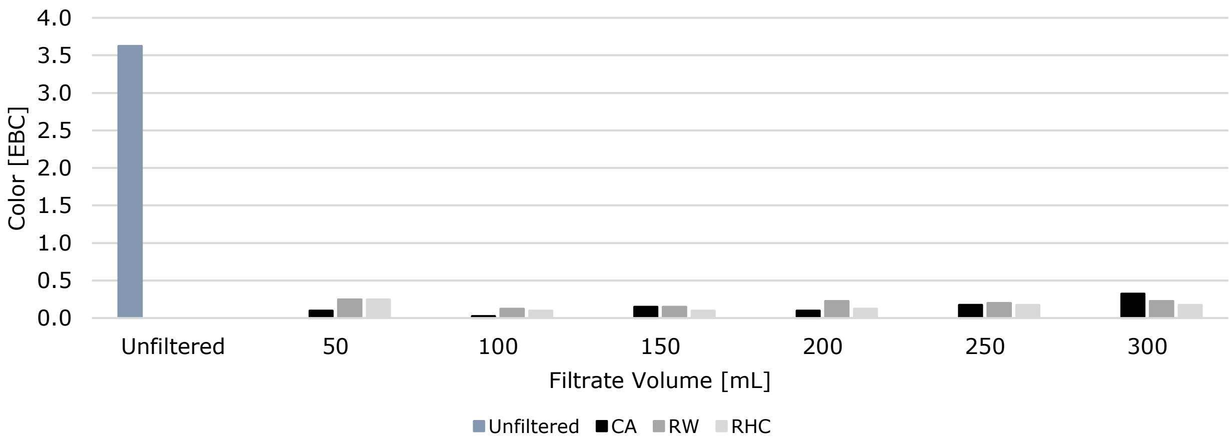

Figure 3: EBC values of the unfiltered hard seltzer, the reference, and the filtration fractions of all trials.

3.2 Production Scale Trials

The initial EBC value of 2.5 was reduced to 0.26 EBC after the filtration with the FILTROX CARBOFIL™ CA. As already shown in the laboratory scale trials, the color was successfully removed by the activated carbon treatment.

The achieved ester values are compared with the unfiltered cider in figure 4. The unfiltered cider exhibited an ethyl decanoate and ethyl dodecanoate concentration of 378.2 µg/L and 18.4 µg/L respectively, whereas the levels in the filtrate was reduced below the limit of quantification (LOQ). A reduction of ethyl hexanoate by 93% and ethyl octanoate by 98% was reported. An initial ethyl butyrate concentration of 263.5 µg/L was measured in the untreated sample. After the filtration, an amount of 233.0 µg/L was reached, resulting in a smaller reduction of 12 %. Overall, the effectiveness on the removal of unwanted flavor compounds from a cider by the treatment with immobilized activated carbon sheets could be documented. Depending on the type and size of the aroma molecules, different concentrations were reached in the final hard seltzer base.

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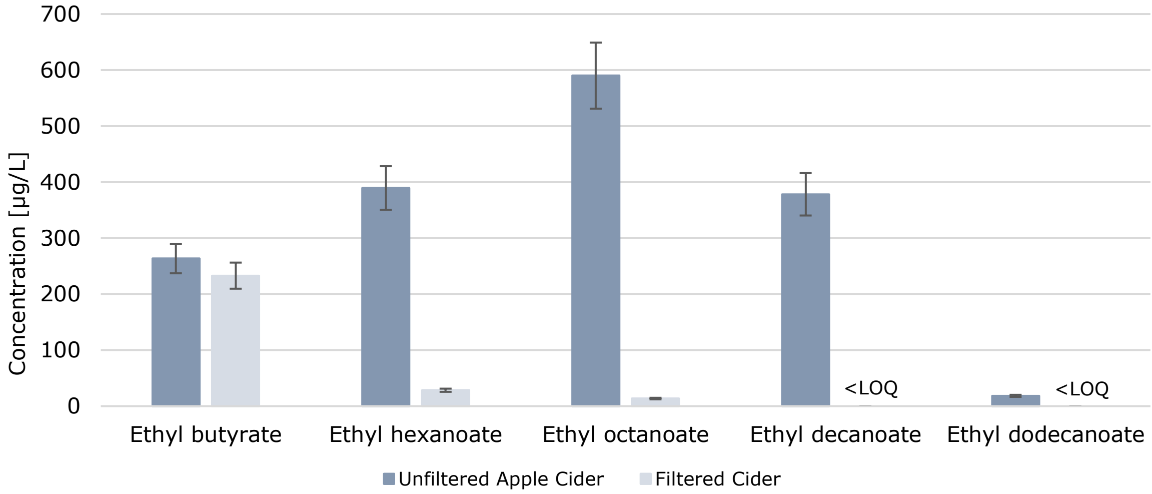

Figure 4: Analysis of Esters after the pilot scale filtration trial. LOQ = Limit of Quantification.

4 Conclusions

Based on the analysis, sufficient color reduction is achieved with only one filtration step. In addition to the short-term tests, long-term tests play a decisive role in determining the maximum capacity of the appropriately used activated carbon filter sheet. This determines the point in time when no longer enough color molecules from the cider can be removed and thus the target values regarding color reduction are no longer met.

The ester content of the unfiltered and the filtered Seltzer were analyzed as these substances have a significant impact on the final aroma. Overall, carbon treatment is an excellent technique for color and odor reduction of a hard seltzer base. The selection of the most suitable type of immobilized activated carbon filter sheet should always be tested on a case by case basis.

Literature

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EBC method color: 9.6 - Colour of Beer: Spectrophotometric Method (IM)

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