

## AFTERTREATMENT OF RAPESEED OIL FUEL TO REDUCE ELEMENT CONTENTS

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**ABSTRACT:** Aftertreatment with added substances, such as bleaching earth, silica gel and citric acid during the cleaning process allows high contents of the deposit and ash-forming elements phosphorus (P), calcium (Ca) and magnesia (Mg) in cold-pressed rapeseed oil fuel, produced at small-scaled oil mills, to be reduced below the limits according to the preliminary standard DIN V 51605. In trials at laboratory scale, rapeseed oil with untypically high contents of phosphorus, calcium and magnesia was treated with ten different added substances available on the market and citric acid at different oil temperatures. Afterwards, it was cleaned by means of centrifugation and filtration and then analysed for relevant parameters according to DIN V 51605. In trials at pilot plant scale, rapeseed oil with contents of phosphorus, calcium and magnesia already below the limits according to the preliminary standard DIN V 51605 was treated with six chosen added substances from the laboratory trials and citric acid. The cleaning process was organized like it is at small-scaled oil mills, using a chamber filter press. The gained oil was then analysed for relevant parameters according to DIN V 51605.

**Keywords:** Rapeseed oil fuel, quality, absorption, adsorbent

### 1 INTRODUCTION AND OBJECTIVE

The use of rapeseed oil fuel for the reliable operation of engines suitable for vegetable oil requires high-quality fuel according to DIN V 51605 [1]. As engine development progresses and more exhaust gas aftertreatment systems are used, the reduction of deposit and ash-forming elements, such as phosphorus, calcium and magnesia in rapeseed oil fuel is becoming more and more important [1]. If the requirements with regard to a lower content of these elements in rapeseed oil fuel are tightened, the current technical equipment of small-scaled oil mills will no longer enable fuel qualities to be produced, which are conform to the limits according to DIN V 51605. Therefore, suitable techniques for the aftertreatment of rapeseed oil would become necessary for this purpose [1].

In small-scaled oil mills, vegetable oil is gained in two process steps: oil pressing and multiple-stage solid/liquid separation by means of sedimentation and/or filtration. In general, the oil does not undergo additional treatment [14].

From the literature, it is known that bleaching earths [2] [8] [13] [15] and other substances, such as diatomaceous earths [9] [10], cellulose [9] [10] [17] and synthetic silica gels [3] [4] [5] [6] [7] [8] [11] [12] [13] [15] [16] are suitable for the elimination of phosphorus, calcium and magnesia from vegetable oils.

The addition of acid (citric acid or phosphoric acid) and water to vegetable oil [14] allows phospholipids and the metal ions associated with them, such as calcium, magnesia, iron and copper to be removed from the oil.

It is the aim of this study to investigate different methods, which could be suitable for small-scaled oil mills to reduce the content of phosphorus, calcium and magnesia in rapeseed oil fuel, produced at small-scaled oil mills, to examine selected techniques with regard to their suitability for application in such plants and their reduction potential. Other quality parameters of rapeseed oil fuel should not be affected by this treatment.

### 2 APPROACH

#### 2.1 Trials at laboratory scale

In batch trials at laboratory scale, ten different adsorptive or absorptive substances which are available on the market as well as citric acid (C) (20 %) were added to cold-pressed rapeseed oil with untypically high contents of phosphorus, calcium and magnesia. Afterwards, the oil was treated at different temperatures and cleaned.

The turbid oil was stored at a constant temperature of 18 °C and under the conditions of permanent homogenisation. For the batch trials, the oil batches including the turbid matter contained in the oil were homogenised to the highest possible degree. With the aid of an electric contact thermometer, the turbid oil was kept at a constant temperature and homogenised by a heating magnetic agitator. When the predetermined temperature was reached, the turbid oil was conditioned at this temperature for the determined period with or without an added substance and with or without citric acid. An overview of the trial variants is given in Table I and Table II.

**Table I:** Description of experimental variants (trials at laboratory scale) depending on concentration of added substances, oil temperature while conditioning, period of conditioning and concentration of added citric acid

	unit	coding of samples at laboratory scale							
		0-25	0-45	0-45-C	0,2-25	0,2-45	1-25	1-45	1-45-C
concentration of added substance	weight-%	0,0	0,0	0,0	0,2	0,2	1,0	1,0	1,0
oil temperature while conditioning	° C	25	45	45	25	45	25	45	45
period of conditioning	min	30	30	30	30	30	30	30	30
concentration of added citric acid	weight-%	0,0	0,0	1,0	0,0	0,0	0,0	0,0	1,0

In those variants where citric acid was added, the acid was first stirred into the oil for two minutes as soon as the turbid oil had reached the desired temperature. Only afterwards was the prepared substance quantity added to the turbid oil-citric acid mixture, which was subsequently conditioned at the set temperature for the determined period.

**Table II:** Added substances applied and coding of experimental variants

abbreviation	term
ZS	Zero Sample
DE	Diatomaceous Earth
SG	Silicagel
CE	Cellulose
BE	Bleaching Earth
BEM	Bleaching Earth Mixture

The oil was cleaned by means of centrifugation (20 min, 5.000 min<sup>-1</sup>) and subsequent vacuum filtration (at about 20 °C) using a filter membrane. The goal was the most precise observation of the predetermined conditioning period possible and hence, the contact duration of turbid oil, added substances and citric acid. If possible, longer contact times of individual added substances in the treated oil due to a longer cleaning process should be excluded. At 3 to 12 mg/kg, the total contamination of the cleaned oils was always kept below the limit of 24 mg/kg according to DIN V 51605.

In order to guarantee constant oil quality over the entire trial period, turbid oil samples were taken from the storage tank at intervals of two weeks. Later, this period was extended to four weeks. These samples were cleaned and the pure oil produced was analysed based on parameters according to DIN V 51605.

The treated and cleaned oils were examined for the parameters phosphorus (DIN EN 14107), calcium and magnesia (E DIN EN 14538), total contamination (DIN EN 12662), acid number (DIN EN 14104), oxidation stability (DIN EN 14112), ash content (DIN EN ISO 6245) and water content (DIN EN ISO 12937). In addition, an ICP element screening was carried out for all samples in order to detect potential contaminations of the pure oil due to the treatment with added substances.

## 2.2 Trials at pilot plant scale

In trials at pilot plant scale, rapeseed oil with contents of phosphorus, calcium and magnesia already below the limits according to the preliminary standard DIN V 51605 was treated with six chosen added substances from the laboratory trials and citric acid at a predetermined temperature and then cleaned. The contents of phosphorus, calcium and magnesia should be reduced close to the detection limit (1 mg/kg). The gained oil was then analysed for relevant parameters according to DIN V 51605.

With these trials, the effectiveness of the chosen added substances and citric acid to reduce the contents of the deposit and ash-forming elements phosphorus, calcium and magnesia in rapeseed oil fuel should be examined as well as the suitability of such techniques for the aftertreatment of rapeseed oil fuel at small-scaled oil mills.

The cleaning process was organized like it is at small-scaled oil mills, using a chamber filter press, to

investigate the maintained special developing effectiveness of diatomaceous earths, celluloses and bleaching earths inside the filter cake for the reduction of the contents of phosphorus, calcium and magnesia in the treated oil. With cleaning the oil by means of centrifugation and filtration at the trials at laboratory scale, this investigation was not possible. Practical experiences with the diatomaceous earths DE1 and DE2, the celluloses CE1 and CE2 and the bleaching earth mixture BEM show differing results to the trials at laboratory scale. Therefore, further investigations with these added substances should be carried out by using a chamber filter press. It was also planned to examine, whether the addition of citric acid to the turbid oil has an influence on the structure of the filter cake, the function of the chamber filter press, the characteristics of filtering the treated oil and the reachable oil quality.

After pressing the rapeseeds, the turbid oil was stored in portions of about 200 liter in barrels until the trials were carried out. By this, homogeneous oil batches including the turbid matter contained in the oil could be supplied for each single trial. For each trial, the turbid oil was filled into a tank and heated to 45 °C under the conditions of permanent homogenisation.

When the predetermined temperature was reached, the turbid oil was conditioned at this temperature for the determined period with or without an added substance and with or without citric acid. An overview of the trial variants is given in Table 2 and Table 3.

In those variants where citric acid was added, the acid was first stirred into the oil for 15 minutes as soon as the turbid oil had reached the desired temperature. Only afterwards was the prepared substance quantity added to the turbid oil-citric acid mixture, which was subsequently conditioned at the set temperature for the determined period of 30 minutes.

The oil was cleaned by means of filtration using a chamber filter press. At 2 to 22 mg/kg, the total contamination of the cleaned oils was always kept below the limit of 24 mg/kg according to DIN V 51605.

Analogous to the trials at laboratory scale, the treated and cleaned oils were examined for the parameters phosphorus (DIN EN 14107), calcium and magnesia (E DIN EN 14538), total contamination (DIN EN 12662), acid number (DIN EN 14104), oxidation stability (DIN EN 14112), ash content (DIN EN ISO 6245) and water content (DIN EN ISO 12937). In addition, an ICP element screening was carried out for all samples in order to detect potential contaminations of the pure oil due to the treatment with added substances.

**Table III:** Description of experimental variants (trials at pilot plant scale) depending on concentration of added substances, oil temperature while conditioning, period of conditioning and concentration of added citric acid

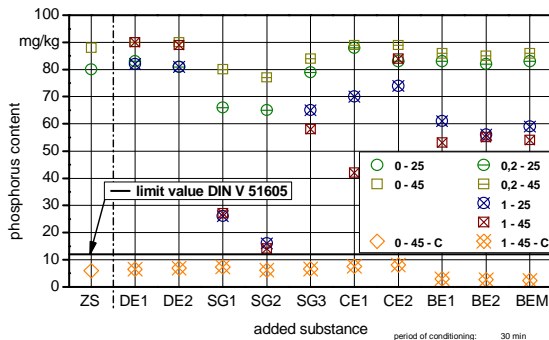
	unit	coding of samples at pilot plant scale			
		0-45	0-45-C	0,5-45	0,5-45-C
concentration of added substance	weight-%	0,0	0,0	0,5	0,5
oil temperature while conditioning	° C	45	45	45	45
period of conditioning	min	30	30	30	30
concentration of added citric acid	weight-%	0,0	0,35	0,0	0,35

### 3 RESULTS

#### 3.1 Trials at laboratory scale

##### a. Influence on the phosphorus content

Given the high initial content, the addition of silica gels SG1 and SG2 (1,0 weight-% each) allowed the phosphorus content of the oil to be reduced significantly as compared with the reference sample, though not below the limit of 12 mg/kg according to DIN V 51605. The results are summarized in Figure 1. By adding cellulose CE1 at a concentration of 1,0 weight-% and an oil temperature of 45 °C, the phosphorus content is reduced by approximately 50 %. Given an added quantity of 1,0 weight-% and an oil temperature of 45 °C, silica gel SG3, bleaching earths BE1 and BE2 and the bleaching earth mixture BEM also reduced the phosphorus content, though to a lesser extent. In this trial series, diatomaceous earths DE1 and DE2 had no or virtually no influence on the phosphorus content. The noticeably positive effect of higher added substance concentration at a higher temperature (SG2, SG3, CE1, BE1, BE2, BEM) does not apply to diatomaceous earths. The addition of 1,0 weight-% of citric acid at an oil temperature of 45 °C allows the phosphorus content to be reduced significantly below the limit of 12 mg/kg according to DIN V 51605. The additional combination of citric acid and added substances seems useful only for bleaching earths BE1 and BE2 as well as the bleaching earth mixture BEM in order to reduce the phosphorus content even more.

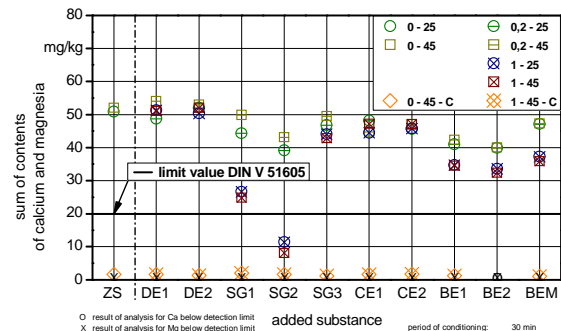


**Figure 1:** Phosphorus content (DIN EN 14107) of rapeseed oil samples after treatment without/with added substances, without/with citric acid (20 %), with variation of oil temperature during conditioning (trials at laboratory scale)

##### b. Influence on the content of calcium and magnesium

In the present series of trials, only the addition of 1,0 weight-% of silica gel SG2 enables the sum of contents of calcium and magnesium to be reduced below the limit of 20 mg/kg according to DIN V 51605. The results of the trial series with regard to the sum of contents of calcium and magnesium are shown in Figure 2. In this trial series, diatomaceous earths DE1 and DE2, celluloses CE1 and CE2 and silica gel SG3 exerted no or virtually no influence on the sum of contents of calcium and magnesium. At a concentration of 1,0 weight-%, silica gel SG1 reduces the sum of contents of calcium and magnesium by about 50 %, though not below the limit of 20 mg/kg according to DIN V 51605. At a concentration of 1,0 weight-%, bleaching earths BE1 and BE2 as well as the bleaching earth mixture BEM enable the sum of contents of calcium and magnesium to be reduced only slightly. The noticeably positive effect of higher added

substance concentration at higher temperatures on the reduction of the element content, which was determined for phosphorus, is less pronounced in the case of calcium and magnesium. Such an effect can only be detected, if silica gels SG1, SG2 and SG3, bleaching earth BE2 and the bleaching earth mixture BEM are used. The addition of 1,0 weight-% of citric acid at an oil temperature of 45 °C allows the sum of contents of calcium and magnesium to be reduced from an initial 52 mg/kg to 1,6 mg/kg. The combination of citric acid and added substances has virtually no additionally reducing effect on the sum of contents of calcium and magnesium. In all analyses of the oils treated with citric acid, the magnesium content was below the detection limit.



**Figure 2:** Sum of contents of calcium and magnesium (E DIN EN 14538) of rapeseed oil samples after treatment without/with added substances, without/with citric acid (20 %), with variation of oil temperature during conditioning (trials at laboratory scale)

##### c. Influence on other quality parameters

The acid number as another quality parameter of rapeseed oil fuel according to DIN V 51605 is virtually not influenced by the treatments carried out. The limit of 2,0 mg KOH/g according to DIN V 51605 is not exceeded in any case.

The oxidation stability is reduced by treatments with added substances except for cellulose CE1 at a concentration of 1,0 weight-%. In some cases, a slight reduction can already be measured at a concentration of 0,2 weight-% (SG2, SG3, BEM).

The added substances exert a wide variety of influences on the water content in the oil. The addition of silica gels SG1 and SG2 to the oil leads to a considerable increase in the water content, which may even exceed the limit of 750 mg/kg according to DIN V 51605. The addition of citric acid also causes a water content increase. A combination of citric acid and diatomaceous earth DE1, silica gels SG1 and SG2, bleaching earths BE1 and BE2 and the bleaching earth mixture BEM as added substances causes the water content to increase even more, even though it remains below the limit according to DIN V 51605.

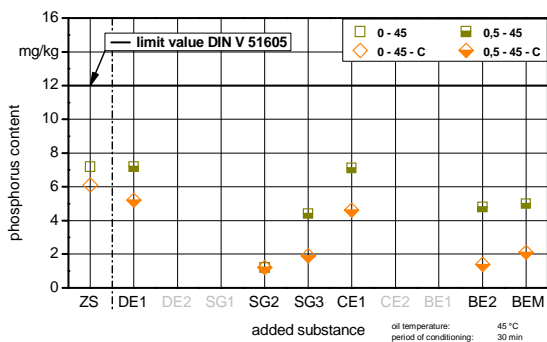
In the reference samples, 3,6 and 4,0 mg/kg of potassium were measured. The treatment of the oil with diatomaceous earths DE1 and DE2 does not influence the potassium content. Silica gels SG1, SG2 and SG3, celluloses CE1 and CE2, and bleaching earths BE1 and BE2 slightly reduced the potassium content. The bleaching earth mixture BEM (concentration: 0,2 weight-%) increased the potassium content considerably from 3,6 to 16 mg/kg and from 4,0 to 14 mg/kg. If a combination of citric acid and added

substances was used, no case occurred where potassium was detected in the oil. The addition of silica gels SG1 and SG2, bleaching earths BE1 and BE2 and the bleaching earth mixture BEM leads to sodium input into the oil, though at a very low level. If the bleaching earth mixture BEM (concentration: 0,2 weight-%) was added, measured sodium contents were 11 mg/kg at an oil temperature of 25 °C and 5,2 mg/kg at 45 °C. If a combination of citric acid and added substances was used, no case was found where potassium was detected in the oil. Other elements, such as iron, copper and zinc, were either not detected at all in the oil, or they were found at concentrations near the detection limit of the measuring technique in the reference sample or after treatment.

### 3.2 Trials at pilot plant scale

#### a. Influence on the phosphorus content

As compared with the reference sample, the addition of silica gel SG2 at a concentration of 0,5 weight-% allowed the phosphorus content of the oil to be reduced from 7,2 mg/kg significantly to 1,2 mg/kg. The results are summarized in Figure 3. By adding silica gel SG3, bleaching earth BE2 and the bleaching earth mixture BEM, a reduction of the phosphorus content was possible, though to a lesser extent with 4,4 mg/kg (SG3), 4,8 mg/kg (BE2) and 5,0 mg/kg (BEM). In this trial series, diatomaceous earth DE1 and cellulose CE1 had no influence on the phosphorus content. The addition of 0,35 weight-% of citric acid at an oil temperature of 45 °C allows the phosphorus content only to be reduced from 7,2 mg/kg to 6,1 mg/kg. The combination of citric acid and added substances allows a more significant reduction of the phosphorus content of the oil. By adding diatomaceous earth DE1 and cellulose CE1 in combination with citric acid, the phosphorus content of the oil could be reduced better, compared to the single addition of citric acid. The combination of citric acid and silica gel SG3, bleaching earth BE2 and the bleaching earth mixture BEM enables the reduction of the phosphorus content of the oil nearly to the reached level with silica gel SG2.

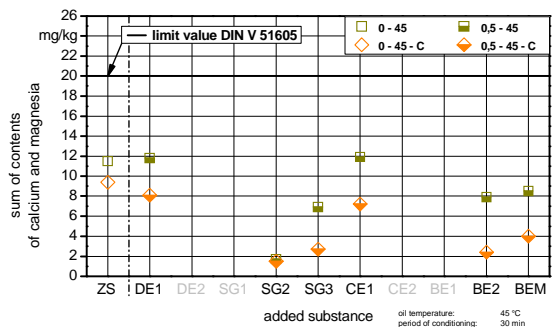


**Figure 3:** Phosphorus content (DIN EN 14107) of rapeseed oil samples after treatment without/with added substances, without/with citric acid (20 %) (trials at pilot plant scale)

#### b. Influence on the content of calcium and magnesium

The practised treatment of the oil with added substances and citric acid leads to similar results for the sum of contents of calcium and magnesium like for the parameter phosphorus content. As compared with the reference sample, the addition of silica gel SG2 at a

concentration of 0,5 weight-% allowed the sum of contents of calcium and magnesium of the oil to be reduced from 11,5 mg/kg significantly to 1,7 mg/kg. The results are shown in Figure 4. By adding silica gel SG3, bleaching earth BE2 and the bleaching earth mixture BEM, a reduction of the sum of contents of calcium and magnesium was possible, though to a lesser extent with 6,9 mg/kg (SG3), 7,9 mg/kg (BE2) and 8,5 mg/kg (BEM). In this trial series, diatomaceous earth DE1 and cellulose CE1 had no influence on the sum of contents of calcium and magnesium. The addition of 0,35 weight-% of citric acid at an oil temperature of 45 °C allows the sum of contents of calcium and magnesium only to be reduced from 11,5 mg/kg to 9,4 mg/kg. The combination of citric acid and added substances allows a more significant reduction of the sum of contents of calcium and magnesium of the oil. By adding diatomaceous earth DE1 and cellulose CE1 in combination with citric acid, the sum of contents of calcium and magnesium of the oil could be reduced better, compared to the single addition of citric acid.



**Figure 4:** Sum of contents of calcium and magnesium (E DIN EN 14538) of rapeseed oil samples after treatment without/with added substances, without/with citric acid (20 %) (trials at pilot plant scale)

#### c. Influence on other quality parameters

The acid number as another quality parameter of rapeseed oil fuel according to DIN V 51605 was apparently negatively influenced by the treatments carried out with added substances and citric acid. The limit of 2,0 mg KOH/g according to DIN V 51605 was already exceeded slightly with the untreated reference sample. It was not possible to verify unambiguously, whether the further increase of the acid number occurred really because of the treatment with added substances and citric acid. Further examinations showed, that the acid number was increasing with an increasing contact time of the turbid matter contained in the uncleaned oil, independent of the treatment with added substances and citric acid.

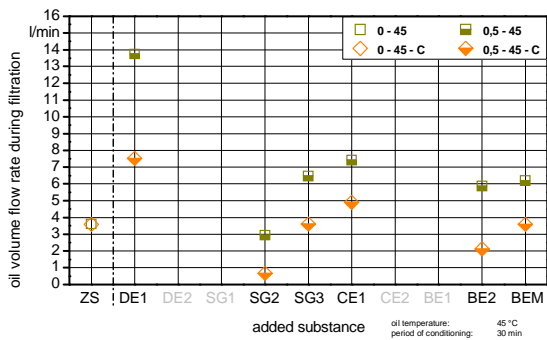
The oxidation stability was not influenced by the treatments carried out with added substances and citric acid.

The addition of silica gel SG2 to the oil lead to a considerable increase in the water content from 676 mg/kg to 971 mg/kg and exceeded the limit of 750 mg/kg according to DIN V 51605. The treatment of the oil with all other added substances reduced the water content in the oil, most significantly with diatomaceous earth DE1. The single addition of citric acid to the oil did not influence the water content in the oil. The combination of citric acid and added substances leads to

an increase in the water content above the limit of 750 mg/kg according to DIN V 51605.

The noticed enrichment with elements in rapeseed oil fuel from the added substances during the trials at laboratory scale was not watched at the trials at pilot plant scale. The analysed contents of elements such as iron, potassium, copper, sodium, silicon and zinc were close to the detection limit.

The oil volume flow rate during filtration was affected negatively by citric acid. The results are summarized in Figure 5. Diatomaceous earth DE1 and cellulose CE1 showed their beneficial effect as a filter aid by improving the oil volume flow rate. Above that, the filter cake was better to remove and the chamber filter press easier to clean.



**Figure 5:** Oil volume flow rate during filtration of rapeseed oil samples after treatment without/with added substances, without/with citric acid (20 %) (trials at pilot plant scale)

#### 4 CONCLUSIONS

At the laboratory scale, the use of added substances allowed the contents of phosphorus, calcium and magnesia in rapeseed oil fuel to be reduced. Silica gel SG2 proved to be particularly suitable for this purpose. However, treatment with added substances (SG1, SG3, BE2, BEM) can also lead to an undesired enrichment with elements such as iron, potassium, copper, sodium, silicon and zinc in the oil. The use of citric acid provides a relatively more noticeable reduction of the phosphorus, calcium and magnesia contents in rapeseed oil fuel. If a combination of added substances and citric acid is used, the observed input of the mentioned elements by added substances can be avoided. In the laboratory, the combination of citric acid with bleaching earths BE1 and BE2 and also with the bleaching earth mixture BEM provided the best results.

During the trials at pilot plant scale, using a chamber filter press to clean the oil, diatomaceous earth DE1 and cellulose CE1 also showed no effect to reduce the contents of phosphorus, calcium and magnesia in rapeseed oil fuel. By using silica gel SG2, the lowest contents of phosphorus, calcium and magnesia in rapeseed oil fuel were analysed. The combination of added substances and citric acid provides a relatively more noticeable reduction of the contents of phosphorus, calcium and magnesia in rapeseed oil fuel. The oil volume flow rate during filtration was affected negatively by citric acid. Diatomaceous earth DE1 and cellulose CE1 showed their beneficial effect as a filter aid by improving the oil volume flow rate.

Differing to the trials at laboratory scale, the treatment with the added substances did not lead to an undesired enrichment with elements in rapeseed oil fuel. The analysed contents of elements such as iron, potassium, copper, sodium, silicon and zinc were close to the detection limit. The influence of method and carrying out of the analysis on the results seems to be quite great.

The positive effect of citric acid to reduce the contents of phosphorus, calcium and magnesia in rapeseed oil fuel was proved at laboratory and pilot plant scale.

Synergetical effects out of the combination of citric acid and added substances for a more noticeable reduction of the contents of phosphorus, calcium and magnesia in rapeseed oil fuel were shown at pilot plant scale with silica gel SG3, bleaching earth BE2 and the bleaching earth mixture BEM.

Concerning the results of the trials at pilot plant scale, citric acid seems to be not suitable for the use at small-scaled oil mills, because the oil volume flow rate during filtration decreased quite heavy by adding citric acid and the chamber filter press was harder to clean.

The negative influence of the used citric acid (20 %) at laboratory and pilot plant scale on the oil volume flow rate during filtration may be avoided by using citric acid (40 %). By this way, less water is brought into the oil.

The beneficial effects of filter aids to the filtering process (higher oil volume flow rate during filtration, more favourable structure of the filter cake, chamber filter press easier to clean) should be used by adding a combination of added substances and citric acid to the oil. Diatomaceous earths are said to be unhealthy when breathing them in, because of the contained crystalline silicates. Celluloses are not harmful to the user's health and can be used as a filter aid.

The effect of the added substance concentration with regard to the reduction of the element contents of phosphorus, calcium and magnesia is often not linear if interaction with other quality-determining characteristics is considered. Therefore, the concentration of added substances, oil temperature while conditioning and period of conditioning must be optimized specifically for each oil to be treated. If citric acid also should be used, the added concentration must be optimized.

#### 5 ACKNOWLEDGEMENTS

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