

OIL RECOVERY FROM SPENT BLEACHING EARTH WITH REFLUX EXTRACTION METHOD USING ACETONITRILE AND N-HEXANE SOLVENT

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Abstract: The growth of the oleochemical industry generated a large amount of spent bleaching earth (SBE) solid waste that causes environmental problems. The SBE solid waste produced is reported at about 600,000 metric tons per year. Recovery of the oil from SBE can be an alternative solution to reduce the negative impact of SBE solid waste. In this study, the recovery of oil from SBE was carried out by the reflux extraction method followed by a study on its properties. The oil recovery process was conducted by using two different solvents (acetonitrile and n-hexane) at different particle sizes (40, 60, 100 mesh) of SBE, solvent-SBE ratios (2:1, 3:1, 4:1), and extraction time (1, 2, 3 hours). Experimental results showed that n-hexane extracted oil with yield ranged from 5 to 14.8%, while acetonitrile extracted oil with yield ranged from 0.68 to 8.4%. Product analysis shows that the oil extracted has a water content of 1.56% and 2.53% for acetonitrile and n-hexane, respectively. The viscosity analysis results show that the oil viscosities were 5.83 cSt (acetonitrile) and 5.09 cSt (n-hexane) which is in reasonable agreement with the Indonesian National Standard for vegetable oil (SNI 7709:2019). The result suggested that n-hexane provided better product yield than acetonitrile as a solvent for oil recovery from SBE. This study provides an alternative method and solution for the oleochemical industry in managing SBE waste.

Keywords: acetonitrile; n-hexane; oil recovery; reflux; spent bleaching earth

Abstrak : Pertumbuhan industri oleokimia menghasilkan limbah padat berupa tanah pemucat (*spent bleaching earth*/ SBE) yang mengakibatkan permasalahan lingkungan. Limbah padat SBE yang dihasilkan dilaporkan sekitar 600.000 ton per tahun. Pemisahan minyak dari SBE dapat menjadi alternatif penyelesaian masalah untuk menurunkan dampak negatif dari limbah SBE. Dalam penelitian ini, *recovery* minyak dari SBE dilakukan dengan metode ekstraksi reflux dan dilanjutkan karakterisasi hasil minyak. Recovery minyak dilakukan dengan dua jenis pelarut (asetonitril and n-heksana) dengan ukuran partikel SBE bervariasi (40, 60, 100 mesh), perbandingan pelarut-SBE (2:1, 3:1, 4:1), dan waktu ekstraksi (1, 2, 3 jam). Hasil penelitian menunjukkan bahwa asetonitril mengekstrak minyak dengan yield 1,56-2,53%, sementara n-heksana berkisar 5-14,8%. Minyak hasil ekstraksi menunjukkan kadar air 1.56% (asetonitril) and 2.53% (n-heksana), serta nilai viskositas sebesar 5.83 cSt (asetonitril) and 5.09 cSt (n-heksana). Minyak hasil

memenuhi SNI Minyak goreng sawit (SNI 7709:2019). Hasil menunjukkan n-heksana memiliki kemampuan lebih baik dibanding asetonitril dalam mengekstrak minyak dari SBE. Hasil penelitian memberikan penyelesaian alternatif dalam menagngani limbah SBE dari industri oleokimia.

Kata kunci : asetonitril; n-heksana; recovery minyak; refluks; SBE

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Introduction

Processing of vegetable or animal oils in Indonesia can produce oleochemicals. Palm oil is a triglyceride so it consists of fatty acid compounds & glycerol. In addition, palm oil belongs to the oleic-linoleic acid group. Carotenoid pigment content (β -carotene) causes palm oil to have a reddish-orange color (Muchtadi & Sugiyono, 2010). Spent bleaching earth (SBE) is a waste product of the palm oil bleaching process, which contains 20-30% weight of palm oil. The amount of SBE produced by the palm oil industry in Indonesia reaches 750,000 tons/year (Nugroho & Singgih, 2021).

Bleaching is a treatment that removes the color substances and other impurities such as fatty acids, gums, trace metals, and phosphatides from crude palm oil. The usual method of this process is the adsorption of the impurities by bringing the oil into contact with a surface-active microporous adsorbent material or bleaching agent (Egbuna et al., 2015). Activated bleaching earth and activated carbon are widely used as adsorbents in the refining industry for the bleaching of edible oil. Adsorptive bleaching is mostly affected by temperature and humidity, but structure and type of adsorbent also play an important role (Raji et al., 2018).

One of the wastes produced by the oleochemical industry or CPO is spent bleaching earth which is a group of waste from processing animal/vegetable fats and derivatives but there is still an oil content of 20 - 40% so it has the potential for the recovery process (Oladosu, 2017). The amount of SBE produced by the palm oil industry in Indonesia reaches 750,000 tons/year. Government regulation number 101 2014 categorizes SBE as hazardous waste with a code B-413. Therefore, the companies must carry out environmental protection and management for SBE. According to the National Vegetable Oil Industry Organization, 17 units of integrated SBE utilization are needed to recover oil (R-oil) and de-oiled Bleaching Earth (OBE) (Nugroho & Singgih, 2021).

SBE is a component that has the main composition of silicon dioxide, aluminum oxide, water bound as well as Ca^{2+} ions, MgO and Fe_2O_3 . The presence of Al^{3+} ions is the cause of the discretion on the surface of the trapping particles and has an impact on the process of adsorption of color substances and adjusting the ratio of aluminum oxide and SiO_2 (Ketaren, 1986). Based on the Regulation of the

Minister (PerMen) LHK No. 10 2020 on Characteristic Test and Determination of Waste Status mentioned that there is a category that shortened the procedure of applying for waste exclusion with one type of waste, namely SBE <3%. This is because SBE waste still has various benefits and can provide 4P benefits (People, Planet, Profit, Prosperity) so it is necessary to use SBE waste that can be done initial research on a laboratory scale. The oil with a high level of free fatty acids found in spent bleaching earth (SBE), which is typically between 17% and 28% by weight, is not removed during the final filter process. Therefore, it must be handled carefully before being disposed of. The main concern with handling SBE from the bleaching of unsaturated vegetable oils is the risk of spontaneous auto ignition, so it is classified as hazardous waste (Fattah et al., 2014). SBE waste can have high economic value if it is properly processed. Based on (Tim Riset PASPI, 2020) shows that after proceeding with solvent extraction, SBE can produce two raw materials, recovered oil (R-Oil) and de-oiled bleaching earth (De-Obe). SBE is also considered a good adsorbent to adsorb dye from aqueous solutions. Based on pore space in their morphology surface and chemical composition, SBE has potential as an adsorbent material (Yulikasari et al., 2022). Research conducted previously (Shehab et al., 2019) that SBE can be extracted using the reflux extraction method and the result shows that it can be used as a dye adsorbent.

The solvent type and the method of oil extraction usually determine the quality of the extracted oil from SBE (Mu & Wang, 2019). Selection of a suitable solvent, based on the organic compounds in SBE, is the most important decision in this process. These compounds in nature are divided into two categories including polar compounds such as pigments, oils, and nonpolar fractions (Merikhy et al., 2018). Regenerated SBE by some organic solvents and the order of the obtained extraction efficiencies were as follows: methyl ethyl ketone > acetonitrile > n-hexane (Merikhy et al., 2018). In their research, the molecular weight of the solvents was discussed as the most effective parameter in the extraction process. With increasing the molecular weight, extraction efficiency decreases (Shahi, 2015). The sample material to solvent volume ratio affects oil yield and density. The best treatment for the ratio of material to solvent volume for sample A is 1:8 with a maximum oil yield of 88.31% (Utami & Nastiti Siswi Indrasti, 2020). SBE can be extracted by several methods such as microwave-assisted extraction (Nugraha et al., 2023), maceration extraction (Sumada, 2022), thermal remediation (Sabour & Shahi, 2018), solvent extraction (Low et al., 2022). A study implied that oil from SBE can be recovered by extraction techniques (Machmudah et al., 2022). There are several options for extracting the residual oil from SBE, these include pressurized carbon dioxide, treating with pressurized hot water, steam extraction, and solvent extraction (Oladosu, 2017). Oladosu (2017) conducted soxhlet extraction using hexane as a solvent and used the reflux method and some variations to know which one produced the best result.

Reflux extraction is the extraction by heating samples and solvents based on their boiling point temperature in a given time using a relatively constant limited quantity of solvents and equipped with condensers. The extraction process can attract samples of compounds effectively and efficiently by solvents (Susanty & Bachmid, 2016). If compared with the maceration method, reflux extraction methods can obtain higher conversion (Nurasiah, 2010). Then when compared with the Soxhlet method, reflux extraction is more efficient and requires less extraction time and solvent (Zhang et al., 2018). The result exhibited that reflux extraction produced the highest yield of extraction (Hartanti & Theeravit, 2018).

Reflux extraction methods can be used in treating waste spent bleaching earth (SBE) so that the goal of implementing the concept of 3R (reduce, recycle, recover) can be achieved. This study was conducted by varying extraction time, particle size, material composition, and solvent. Many solvents are usually used in SBE oil extraction namely benzene, toluene, xylene, and n-hexane (Abdelbasir et al., 2023). While acetonitrile has been also widely used as a solvent for oil extraction for upgrading the light cycle oil (Olar et al., 2021). N-hexane is the result of refining crude oil. N-hexane composition and fractions are affected by the oil source. At 60 °C to 70 °C, typically 50% of the isomer chain will boil. All of the hexane's isomers are frequently used as inert organic solvents because it is non-polar. Usually, it is used for oil extraction from seeds. Oil extraction doesn't need high temperatures and heat (Utomo, 2016). As reported by Naser et al. (2021), n-hexane as a solvent produces better residual oil if it is desired for biodiesel production as it gives a higher FAME (fatty acid methyl esters) composition. It was noted that the use of hexane-extracted SBE to remove dyestuff from wastewater was a program aiming at cleaning up the environment by the use of SBE as it was inexpensive and easily available (Mu & Wang, 2019). In addition to producing new fuel, SBE recovery is important to reduce the quantity of SBE.

SBE could serve as a useful adsorbent for the removal of organic basic dyes after being extracted with hexane. Thus, this approach can be an alternative solution to develop methods to manage waste from spent bleaching earth. This research is an effort to recover oil by utilizing waste spent bleaching earth from oleochemical production. The recovery process was conducted by reflux method using n-hexane and acetonitrile. The results would be compared and investigated to study the effect of different solvents on oil properties.

Materials And Method

Materials

Spent bleaching earth (SBE) was obtained from one of the oil industries in Indonesia as a result of the bleaching process. Acetonitrile (95%) and n-hexane (95%), as solvents were obtained from Merck. For sample analysis, KOH and PP indicators were used for testing of free fatty acid content and acid number.

Methods

The experimental procedure comprises steps i.e screening of SBE, extraction, distillation, and oil product analysis. The procedure is depicted in Figure 1.

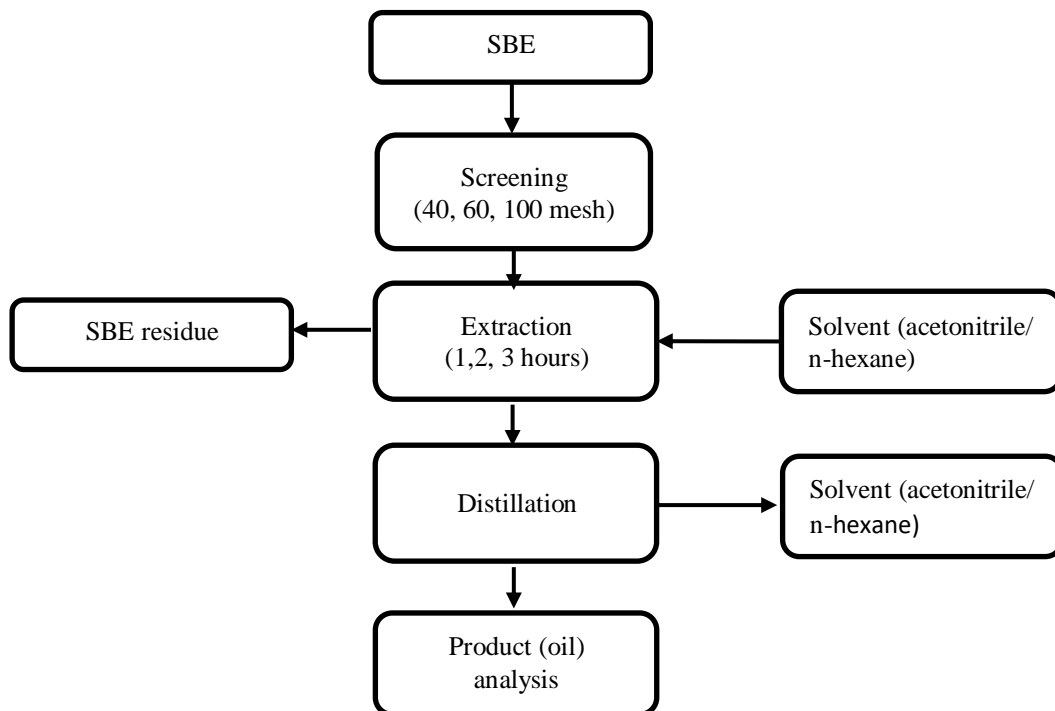


Figure 1. Flowchart of the experimental procedure

Screening of SBE

At the material preparation stage, the SBE sample is first manually grinded and then screened to obtain particle sizes of 40, 60, and 100 mesh. Meanwhile, for sizes greater than >40 mesh, grinding is carried out manually to obtain maximum results. The size 40–100 mesh was selected, that SBE with a size between 40 and 100 mesh is appropriate for use as an adsorbent (Amelia & Maryudi, 2019).

Extraction

Extraction was carried out by mixing a certain volume of solvent with 25 grams of SBE with the various ratios of solvent/SBE of 2:1 (50 ml/25 g SBE); 3:1 (75 ml/25 g SBE); and 4:1 (100 ml/25 g SBE) in each sample using reflux methods. During the extraction process, the acetonitrile/n-hexane solvent was added with variations of 50, 75, and 100 ml and time variations of 1, 2, and 3 hours. SBE after the screening was used as feed in the reflux extraction process with the extraction temperature following the boiling point of the solvent. The result of the extraction process was a mixture of oil-solvent that would be separated to determine the quantity of oil.

Distillation

The separation of oil from solvent in the mixture was conducted using a batch distillation process. The distillation process was set at boiling temperature of each solvent (82°C for acetonitrile and 60°C for n-hexane) for 1 hour. The oil-solvent mixture was boiled up and let solvent evaporate. The residue obtained was oil that was free from solvent. The oil products then were characterized to investigate their properties.

Oil Product Analysis

The test for investigating oil characteristics comprised volume of oil, viscosity, water content, and free fatty acids (FFA). The oil extracted was then analyzed for its water content and FFA levels based on SNI 01-355S-1998. The water content test was carried out using the oven for 1 hour at a temperature of 105°C. The FFA level test was done by adding 95% ethanol and heating the mixture at 70°C for 20 minutes. The FFA level was calculated by titration methods using KOH 0.1 N and PP indicator until it turned pink. Furthermore, the viscosity test was also conducted based on ASTM D445 by heating the oil at a temperature of 40°C and then measured using a viscometer based on the oil drop from the upper limit to the limit of the viscometer.

Results And Discussion

This study compared the performance of solvents: n-hexane and acetonitrile in extracting oil from SBE. Both types of solvents were used during the process of oil uptake by varying three parameters: the size of SBE (40, 60, 100 mesh), the ratio of solvent: SBE (2:1, 3:1, 4:1) and extraction time (1, 2, 3 hours).

Effect of SBE size

Figure 2 shows the results on the effect of SBE size on oil yield for both solvents. It can be seen that the size of SBE particles significantly affects the product yield. Higher mesh size generated higher oil yield from the oil recovery process. The highest oil yield was obtained from the smallest SBE particle size or the highest mesh size (100 mesh). This result follows results from previous research (Kuuluvainen et al., 2015) which reported that the smaller the particle size of SBE, the more oil will be produced because the contact field between SBE and solvent will also be wider. With acetone as the solvent, the oil yield was 24.14%, whereas with n-hexane, it was 15.37%.

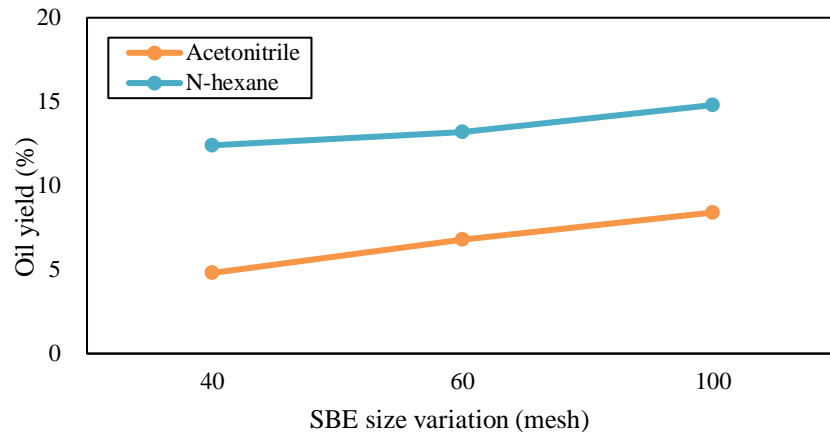


Figure 2. Effect of SBE size variation on oil yield for different solvent

Figure 2 also shows that extraction using n-hexane as solvent obtained higher oil yield than acetonitrile. Prior research reported that the product yield of SBE extraction using n-hexane solvent ranged from 16.45 to 17.9% (Kuuluvainen et al., 2015). This result is the following results from research conducted by Nezhbahadori et al. (2018) and Utomo (2016) that reported non-polar solvent is better than polar solvent during oil recovery using solvent extraction. Additionally, the solubility of n-hexane in oil is stronger than in acetonitrile. This is why the yield using n-hexane as a solvent is better than acetonitrile. In terms of product (oil) yield and quality, the use of n-hexane in solvent extraction provided better results compared to mechanical extraction. The oil concentration of the residues after solvent extraction is less than 3%, while the concentration in press cakes is 8-9% at the same mild operating temperature (Li et al., 2014).

Effect of solvent: SBE ratio

Figure 3 shows the yield of oil produced on various compositions of solvent and SBE from each solvent during oil extraction. The data presented in Figure 3 were taken at an SBE size of 100 mesh and an extraction time of 3 hours. The result reveals that the production of oil increases following the higher ratio of solvent used in the extraction process.

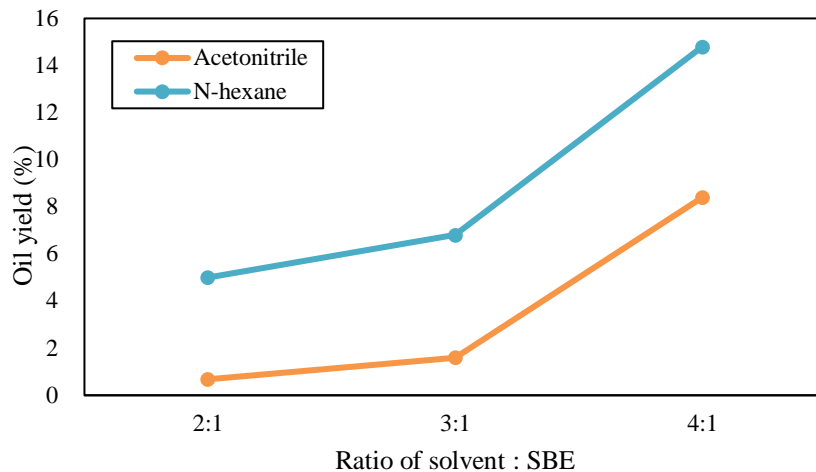


Figure 3. Effect of solvent and material ratio on oil yield for different solvent

Increasing the ratio of solvent and SBE from 2:1 to 3:1 slightly improved the yield. However, by increasing the ratio by double (from 2:1 to 4:1), the oil yield stepped up significantly from 5% to 14.8% for n-hexane and from 0.68% to 8.4% for acetonitrile. The highest oil yield was obtained at the highest ratio of solvent and SBE, which was 4:1. The same result was reported by Elwardany et al., (2023) Increasing the solvent to SBE ratio from 4:1 to 5:1 increased oil yield, because the concentration gradient between the solid and solvent becomes larger which acts as a driving force in the mass transfer process.

In the previous study (Melwita et al., 2014), it was stated that a high amount of solvent in the oil recovery process means that the number of dissolved compounds was abundantly present in the mixture. This will lead to a higher extraction rate so that more product is generated.

Effect of extraction time

The effect of extraction time variation on oil yield for different solvents at a solvent-SBE ratio of 4:1 and SBE size of 100 mesh was illustrated in Figure 4.

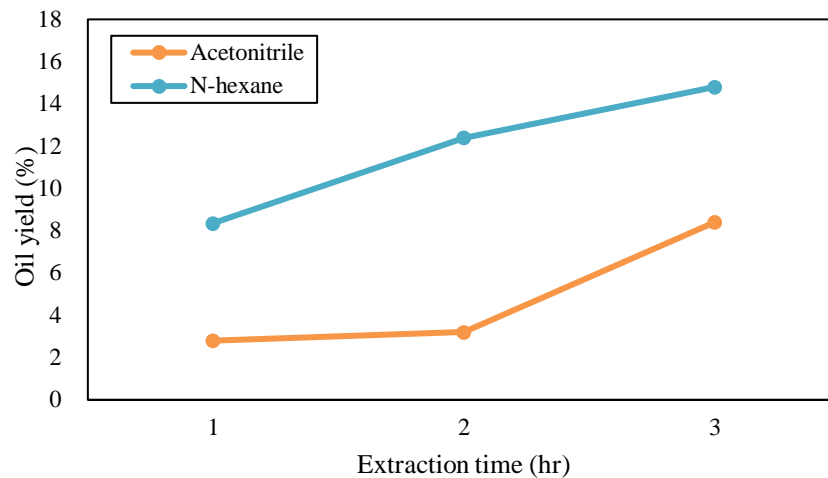


Figure 4. Effect of extraction time on oil yield for different solvents

Figure 4 indicates that extraction time affects the oil yield, as longer extraction time will generate more product from the oil recovery process. It can be seen that the optimum time for conducting oil recovery from SBE in this study was 3 hours, which yielded oil 4 times higher 2.8 % to 8,4% (for acetonitrile solvent) and almost 2 times higher 8.36% to 14.8% (for n-hexane solvent). The time of extraction becomes one of the factors that affect the extraction results, because the length of extraction time will have an impact on the length of contact of the material with the solvent, so the longer the time is directly proportional to the time of contact of the material with the solvent which causes more solutes contained in the solids to dissolve in the solvent (Nasir et al., 2009; Zulqarnain et al., 2021).

Product characterization

Oil produced from solvent extraction was characterized using standard parameters based on the Indonesian National Standard for Vegetable Oil (SNI 7709:2019) and The Roundtable on Sustainable Biomaterials (RSB, 2008). The comparison of each parameter value is shown in Table 1.

Table 1. Result of product characterization

Parameter	Solvent		Vegetable Oil	
	Acetonitrile	n-hexane	(RSB, 2008)	Indonesian National Standard (SNI 7709:2019)
Water content (%)	1.56	2.53	<1	Max 0.1
FFA (%)	14.51	8.96	Standard FFA (<5%) High FFA (5-20%)	0.3
Viscosity (cSt)	5.83	5.03	16.65	2.3 - 6.0

Table 1 shows the results of oil characterization by analysis of water content, free fatty acids (FFA), and viscosity. It can be seen that the only parameter which was following the standards was viscosity. The product from the oil recovery process in this study has a viscosity of 5.03 and 5.83 for acetonitrile solvent and n-hexane solvent, respectively. These values have not met the vegetable oil standards of the Indonesian National Standard (SNI 7709:2019). If the viscosity is too high, it can impact the process of atomization of fuel and air leading to evaporation, so the combustion becomes imperfect (Laila, 2017).

Conclusion

Spent bleaching earth (SBE) was successfully regenerated to remove oil content by reflux extraction using two solvents, acetonitrile and n-hexane. The smaller size of SBE gives a higher oil yield. The higher solvents-SBE ratio results in a higher yield of oil, and the longer extraction time results in a higher oil yield. The optimum condition for oil extraction from SBE was obtained at an SBE size of 100 mesh, solvent-SBE ratio of 4:1, and extraction time of 3 hours. It is also found that n-hexane extraction performance (oil yield of 14.8%) is better than acetonitrile (oil yield of 8.4%). The oil properties do not meet the vegetable oil standard of the Indonesian National Standard (SNI 7709:2019). It is suggested to conduct further treatment for oil product to improve its quality.

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