

RESPONSE SURFACE METHODOLOGY (RSM)
UNTUK OPTIMASI PROSES *RECOVERY OIL* DARI
SPENT BLEACHING EARTH

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TESIS

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PROSES RECOVERY OIL DARI SPENT BLEACHING EARTH***

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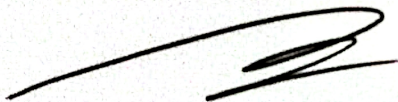
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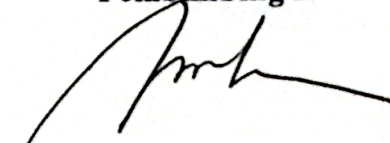
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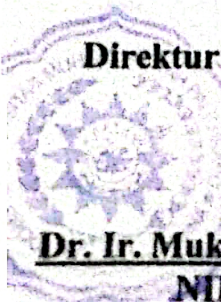
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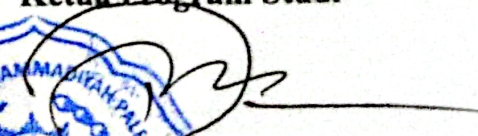
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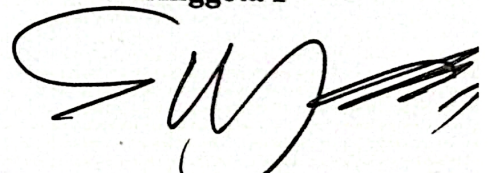
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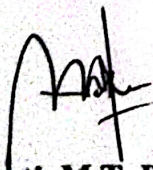
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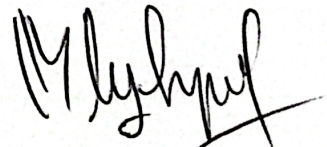
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M. Julian Saputra Jaya

RESPONSE SURFACE METHODOLOGY (RSM) FOR PROCESS OPTIMIZATION OF OIL RECOVERY FROM SPENT BLEACHING EARTH

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ABSTRACT

Spent Bleaching Earth (SBE) is a solid waste generated from the bleaching process in the refining of Crude Palm Kernel Oil (CPKO), which still contains residual oil of approximately 20–40%, giving it potential economic value while also posing environmental risks if not properly managed. This study aims to optimize the oil recovery process from SBE using a reflux extraction method with technical-grade n-hexane solvent through a Response Surface Methodology (RSM) approach based on Central Composite Design (CCD), as well as to evaluate the effect of extraction temperature and time on oil yield and quality. SBE samples were initially characterized using GC–MS and XRF to determine the composition of residual oil and its mineral matrix. The extraction process was carried out with a material-to-solvent ratio (w/v) of 1:8 at a temperature range of 62–66°C and an extraction time of 120–240 minutes with a total of 13 experimental runs. The response variables analyzed included oil yield, moisture content, iodine value, and free fatty acid (FFA) content. The RSM optimization results indicated that the optimum conditions were achieved at approximately 63.47°C and 176.06 minutes, producing an oil yield of 19.08% with acceptable quality (low moisture content, controlled FFA, and stable iodine value). Technical-grade n-hexane proved effective in extracting the lipid fraction from the SBE matrix due to its non-polar nature, low viscosity, and ease of recovery. Extraction temperature and time had significant and non-linear effects on oil yield and quality, with the quadratic effect of temperature being the dominant factor. These findings demonstrate that the application of RSM can provide efficient, economical, and sustainable operating conditions for utilizing SBE as an alternative oil source, supporting circular economy principles and more environmentally friendly B3 waste management.

Keywords: *Spent Bleaching Earth (SBE); oil recovery; technical-grade n-hexane; reflux extraction; Response Surface Methodology (RSM); Central Composite Design (CCD); process optimization; oil yield; hazardous waste (B3); circular economy.*

RESPONSE SURFACE METHODOLOGY (RSM) UNTUK OPTIMASI PROSES RECOVERY OIL DARI SPENT BLEACHING EARTH

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ABSTRAK

Spent Bleaching Earth (SBE) merupakan limbah padat hasil proses pemucatan (*bleaching*) pada pemurnian *Crude Palm Kernel Oil (CPKO)* yang masih mengandung residu minyak sekitar 20–40% sehingga berpotensi bernilai ekonomi sekaligus menimbulkan risiko lingkungan apabila tidak dikelola dengan tepat. Penelitian ini bertujuan untuk mengoptimalkan proses *recovery oil* dari SBE menggunakan metode ekstraksi refluks dengan pelarut n-heksana teknis melalui pendekatan *Response Surface Methodology (RSM)* berbasis *Central Composite Design (CCD)*, serta mengevaluasi pengaruh suhu dan waktu ekstraksi terhadap rendemen dan mutu minyak yang dihasilkan. Sampel SBE dikarakterisasi awal menggunakan GC–MS dan XRF untuk mengetahui komposisi minyak residu dan matriks mineralnya. Proses ekstraksi dilakukan dengan rasio bahan/pelarut (b/v) 1:8 pada rentang suhu 62–66°C dan waktu 120–240 menit dengan total 13 *running* percobaan. Variabel respon yang dianalisis meliputi rendemen minyak, kadar air, bilangan iod, dan kadar asam lemak bebas (FFA). Hasil optimasi RSM menunjukkan bahwa kondisi optimum dicapai pada suhu sekitar 63,47°C dan waktu 176,06 menit, menghasilkan rendemen minyak 19,08% dengan mutu yang memenuhi kriteria (kadar air rendah, FFA terkendali, dan bilangan iod stabil). Pelarut n-heksana teknis terbukti efektif mengekstraksi fraksi lipid dari matriks SBE karena sifatnya yang non-polar, berviskositas rendah, dan mudah didaur ulang. Suhu dan waktu ekstraksi berpengaruh signifikan dan bersifat non-linear terhadap rendemen dan kualitas minyak, dengan efek kuadratik suhu sebagai faktor dominan. Temuan ini menunjukkan bahwa penerapan RSM mampu memberikan kondisi operasi yang efisien, ekonomis, dan berkelanjutan untuk pemanfaatan SBE sebagai sumber minyak alternatif, mendukung prinsip ekonomi sirkular dan pengelolaan limbah B3 yang lebih ramah lingkungan.

Kata Kunci : *Spent Bleaching Earth (SBE)*; *recovery oil*; n-heksana teknis; ekstraksi refluks; *Response Surface Methodology (RSM)*; *Central Composite Design (CCD)*; optimasi proses; rendemen minyak; limbah B3; ekonomi sirkular.

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BAB I

PENDAHULUAN

A. Latar Belakang Penelitian

Minyak inti sawit, yang dalam istilah internasional dikenal sebagai *Crude Palm Kernel Oil* (CPKO), adalah minyak nabati yang diperoleh melalui proses ekstraksi dari biji atau inti (*kernel*) buah kelapa sawit (*Elaeis guineensis*), yang merupakan bagian terpisah dari daging buah sawit (Paramitha & Ekawati, 2022). Indonesia menempati posisi strategis sebagai salah satu produsen utama minyak kelapa sawit di tingkat global. Minyak inti sawit mentah banyak diminati oleh berbagai negara di kawasan Asia dan Eropa sebagai komponen dasar dalam produksi berbagai produk, seperti minyak goreng cair dan padat, margarin, serta berbagai produk dalam industri oleokimia, termasuk sabun dan kosmetik, maupun dalam sektor bioenergi seperti biodiesel. Tingginya utilitas minyak sawit dalam berbagai sektor industri telah mendorong peningkatan permintaan secara signifikan, khususnya dalam dua dekade terakhir (Lubis et al., 2022).

Crude Palm Kernel Oil (CPKO) dilakukan *pretreatment* sebelum diproses selanjutnya. *Pretreatment* ini dilakukan dengan maksud untuk menghilangkan impuritas yang masih terkandung dalam minyak inti sawit, seperti asam lemak bebas, pigmen warna, fosfatida, dan gum yang dapat mengganggu proses selanjutnya [(Nidzam et al., 2022);(Haryanti et al., 2024)]. Salah satu proses *pretreatment* adalah proses pemucatan atau *bleaching*. Proses *bleaching* pada saat *oil pretreatment* menjadi *treated oil* dilakukan dengan penambahan *bleaching earth* melalui proses adsorpsi sehingga minyak terolah akan lebih pucat (Rosli et al., 2022).

Pada tahap pemucatan dalam proses pengolahan minyak sawit, dihasilkan residu berupa *Spent Bleaching Earth* (SBE), yaitu tanah pemucat bekas yang masih mengandung sejumlah minyak teradsorpsi. Untuk memisahkan SBE dari minyak yang telah mengalami proses *bleaching*, digunakan sistem filtrasi *Niagara Filter*. Sistem ini tidak hanya berfungsi sebagai pemisah, tetapi juga mempersiapkan SBE untuk tahap pengolahan lanjutan. Salah satu proses lanjutan yang penting adalah

steam blowing, yaitu teknik pemisahan minyak dengan menggunakan tekanan uap tinggi untuk mengekstrak sisa minyak dari dalam struktur pori SBE [(Lim, 2022); (Manurung et al., 2024)]. Metode *steam blowing* terbukti efektif dalam meningkatkan efisiensi pemisahan, karena tekanan tinggi dari uap mampu mendesak keluar minyak yang terperangkap dalam partikel SBE. Penting untuk menyoroti bahwa limbah *Spent Bleaching Earth* (SBE) masih mengandung minyak residu dalam jumlah yang signifikan, yakni sekitar 20 hingga 40 persen dari bobot keringnya [(Yuliana et al., 2021); (Panumonwatee et al., 2021)].

Berdasarkan Peraturan Pemerintah (PP) Nomor 101 Tahun 2014 tentang Pengelolaan Limbah Bahan Berbahaya dan Beracun (B3), *Spent Bleaching Earth* (SBE) diklasifikasikan sebagai limbah B3 dengan kode B413. Klasifikasi ini menegaskan bahwa SBE memiliki potensi membahayakan lingkungan dan kesehatan manusia apabila tidak dikelola secara tepat. Sebagai residu padat hasil dari proses pemucatan minyak sawit, SBE mengandung senyawa-senyawa kimia teradsorpsi, termasuk minyak terdegradasi dan senyawa logam berat, yang dapat mencemari lingkungan apabila dibuang sembarangan (Nadhifah & Putri, 2024). SBE (*Spent Bleaching Earth*) memiliki kandungan minyak yang cukup tinggi sekitar 20%-30% (Parkoso et al., 2024). SBE merupakan residu padat yang dihasilkan selama proses refining *Crude Palm Kernel Oil* (CPKO) dalam industri oleokimia. Limbah ini masih mengandung sisa-sisa minyak serta sejumlah logam seperti silika, aluminium oksida, besi oksida, magnesium, dan air. Kandungan tersebut menjadikan SBE sebagai limbah yang bersifat beracun dan berbahaya apabila tidak ditangani atau dikelola secara tepat (Restu et al., 2023).

Jumlah minyak yang masih terkandung dalam SBE dapat diekstraksi dan diolah lebih lanjut sebagai bahan baku dalam industri oleokimia maupun produksi biodiesel. Pemanfaatan ini berpotensi meningkatkan output industri terkait tanpa mengurangi ketersediaan *Crude Palm Kernel Oil* (CPKO) untuk kebutuhan pangan. Selain itu, pengolahan ini juga memberikan solusi terhadap potensi pencemaran terhadap lingkungan yang disebabkan oleh limbah minyak tersebut (Taslim et al., 2022a).

Beberapa diantaranya penelitian terdahulu yang berkaitan dengan *recovery oil* dari *spent bleaching earth* seperti penelitian (Maryudi et al., 2024), terkait *recovery oil* dari SBE dilakukan ekstraksi secara refluks menggunakan pelarut acetonitril maupun n-heksana mampu menghasilkan rendemen minyak dengan yield 5-14,8% untuk pelarut n-heksana dan yield 0,68-8,4% untuk pelarut acetonitril. Hasil penelitian tersebut menunjukkan bahwa menggunakan pelarut n-heksana lebih efektif dibandingkan dengan pelarut acetonitril dalam mengekstrak minyak dari *Spent Bleaching Earth*. Selain itu, penelitian yang dilakukan oleh (Muslich et al., 2020), terkait proses ekstraksi minyak dari *Spent Bleaching Earth* (SBE) dilakukan menggunakan metode refluks dengan pelarut *n-heksana*, melalui variasi rasio antara massa bahan terhadap volume pelarut (b/v) sebesar 1:4, 1:6, dan 1:8. Hasil penelitian tersebut menunjukkan bahwa menggunakan ekstraksi secara refluks dengan n-heksana sebagai pelarut lebih efektif untuk menghasilkan rendemen minyak tertinggi dari SBE pada rasio bahan/pelarut (b/v) 1:8.

Mengingat pentingnya pengolahan SBE untuk penelitian lebih lanjut dalam meningkatkan *recovery oil* dengan *solvent extraction* menggunakan jenis pelarut n-heksan teknis. Pemilihan pelarut n-heksan teknis didasarkan pada kesamaan tingkat kepolaran antara pelarut dan minyak, yang memungkinkan terjadinya interaksi yang lebih efektif dalam proses ekstraksi. Selain itu, n-heksana teknis memiliki titik didih yang cukup rendah, sehingga mempermudah proses pemisahan antara fraksi minyak dan pelarut, serta mengurangi kemungkinan terjadinya degradasi termal pada komponen-komponen sensitif dalam minyak. Selama beberapa dekade, n-heksan teknis telah digunakan secara luas sebagai pelarut utama dalam industri, khususnya dalam ekstraksi minyak nabati, karena kemampuannya yang tinggi dalam mengekstraksi residu minyak secara efisien (Muslich et al., 2020). Studi ini tidak hanya memperluas pemahaman terhadap mekanisme ekstraksi minyak dari limbah industri, tetapi juga menyediakan landasan ilmiah yang solid untuk optimalisasi proses pemanfaatan limbah padat dalam industri pengolahan minyak sawit secara lebih ekonomis dan berkelanjutan [(Ajayi et al., 2024);(Suardi et al., 2022)]. Pemanfaatan kembali limbah SBE dalam penelitian ini berkontribusi terhadap pengurangan volume limbah yang dibuang, sekaligus mendukung

penerapan prinsip ekonomi sirkular serta pengelolaan limbah B3 yang berkelanjutan. Tidak seperti penelitian sebelumnya yang umumnya menggunakan pelarut laboratorium berkualitas tinggi, studi ini memanfaatkan n-heksan teknis yang lebih ekonomis dan berpotensi ramah lingkungan, meskipun efektivitasnya masih belum banyak diteliti [(Mulyadi et al., 2023a);(Qodim et al., 2023)]. Selain itu, penelitian ini mengajukan hipotesis bahwa variabel suhu dan waktu ekstraksi berkontribusi signifikan terhadap peningkatan yield minyak dari SBE, dengan kecenderungan meningkat hingga mencapai nilai optimum yang dapat diprediksi secara kuantitatif melalui pendekatan *Response Surface Methodology* (RSM). Penerapan pendekatan *Response Surface Methodology* (RSM) memungkinkan identifikasi kondisi optimum proses ekstraksi secara sistematis, sekaligus meminimalkan jumlah percobaan yang diperlukan tanpa mengorbankan keakuratan model statistik yang dihasilkan.

B. Rumusan Masalah

Mengacu pada isu-isu yang telah diidentifikasi dalam latar belakang, penelitian ini dirancang untuk merumuskan permasalahan sebagai berikut:

1. Bagaimana pendekatan *Response Surface Methodology* (RSM) berbasis *Central Composite Design* (CCD) dapat digunakan untuk memprediksi dan mengoptimasi kondisi ekstraksi?
2. Bagaimana pengaruh pelarut n-heksan teknis dalam mengekstrak komponen minyak dari *Spent Bleaching Earth*?
3. Bagaimana pengaruh suhu dan waktu ekstraksi terhadap yield minyak yang dihasilkan?

C. Tujuan Penelitian

Tujuan utama dari pelaksanaan penelitian ini dapat dijabarkan sebagai berikut:

1. Menerapkan metode RSM berbasis CCD untuk memprediksi dan mengoptimasi kondisi ekstraksi (suhu dan waktu) guna memperoleh rendemen maksimum dengan mutu minyak yang memenuhi standar.

2. Menentukan pengaruh pelarut n-heksan teknis dalam mengekstrak komponen minyak dari *Spent Bleaching Earth*.
3. Menentukan pengaruh suhu dan waktu ekstraksi terhadap yield minyak yang dihasilkan.

D. Kegunaan dan Kontribusi Penelitian

Penelitian ini diharapkan mampu menyumbangkan nilai tambah bagi penulis, masyarakat luas, dan institusi pendidikan, antara lain:

1. Memberikan kontribusi ilmiah dalam bidang teknik kimia, khususnya pada kajian proses ekstraksi serta pemanfaatan limbah padat hasil industri.
2. Menjadi tambahan sumber referensi akademik terkait penerapan dan analisis *Response Surface Methodology* (RSM) dalam ranah teknik kimia.
3. Menyediakan rujukan ilmiah bagi peneliti lain dalam mengembangkan metode pemanfaatan *Spent Bleaching Earth* (SBE) secara lebih efisien dan ramah lingkungan.
4. Menghasilkan nilai tambah dari limbah industri, yang pada akhirnya dapat menekan biaya produksi sekaligus menghasilkan produk dengan potensi nilai ekonomi.
5. Mendukung penerapan konsep *zero waste* pada industri pengolahan minyak nabati melalui pengelolaan limbah yang lebih berkelanjutan.
6. Berkontribusi terhadap pengembangan energi terbarukan serta penyediaan bahan baku alternatif bagi industri kimia.

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