

RINGKASAN

Pengaruh Jenis Pereaksi terhadap Persentase Hasil Transformasi Safrol [Hasil Isolasi dari Minyak Lawang (*Cinnamomum cullilawan*, BL)] menjadi Sesamol

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Dewasa ini sesamol, banyak digunakan sebagai antioksidan. Disamping itu, banyak juga digunakan sebagai bahan dasar dalam pembuatan obat-obatan seperti ayapin. Senyawa tersebut merupakan senyawa alami yang terdapat dalam minyak wijen.

Adanya gugus metilendioksi, fenil dan hidroksil pada struktur sesamol, memungkinkan untuk mé sintesisnya dari bahan dasar alami yang lain yakni dari safrol hasil isolasi dari minyak lawang (*Cinnamomum cullilawan*, BL). Pada proses transformasi tersebut diperlukan beberapa tahapan reaksi, mulai dari isomerisasi, oksidasi serta substitusi.

Untuk itu, telah dilakukan penelitian tentang pengaruh jenis pereaksi terhadap persentase hasil transformasi safrol yang diperoleh dari minyak lawang (*Cinnamomum cullilawan*, BL) menjadi sesamol. Minyak lawang yang digunakan dalam penelitian ini adalah cap X yang diperoleh dari Ambon. Penelitian ini bertujuan untuk menentukan jenis pereaksi yang dapat memberikan persentase hasil reaksi yang tertinggi dalam transformasi safrol menjadi sesamol.

Untuk mencapai tujuan tersebut, diperlukan terlebih dahulu pemisahan safrol dari minyak lawang. Pemisahan dilakukan dengan cara distilasi fraksi pengurangan tekanan. Selanjutnya dilakukan transformasi safrol menjadi isosafrol menggunakan lima macam pereaksi yakni *t*-BuOK/DMSO, KOH/*n*-BuOH, KOH/CH₂OHCH₂OH, NaOH/CH₂OHCH₂OH, KOH/C₂H₅OH. Transformasi isosafrol menjadi piperonal menggunakan tiga macam pereaksi yaitu: Na₂Cr₂O₇/H₂SO₄/H₂O, Na₂Cr₂O₇/H₂SO₄/H₂O/asam sulfanilat serta KOH/DMSO-nitrobenzena. Sedangkan transformasi piperonal menjadi sesamol dengan pereaksi MCPBA-CH₂Cl₂/KOH – CH₃OH dan H₂O₂/NaOH.

Pemurnian hasil dilakukan dengan distilasi pengurangan tekanan dan kolom kromatografi, sedangkan uji kemurnian hasil dilakukan dengan kromatografi lapis tipis dan penentuan sifat-sifat fisik seperti indeks bias, berat jenis dan titik leleh. Selanjutnya struktur diidentifikasi dengan spektrofotometer inframerah, resonansi magnetik inti ¹H dan ¹³C serta spektrometer massa.

Hasil penelitian menunjukkan bahwa safrol yang diperoleh dari minyak lawang sebesar 7,05%. Untuk reaksi isomerisasi safrol menjadi isosafrol, pereaksi *t*-BuOK/DMSO memberikan persentase hasil yang tertinggi yakni sebesar 97%. Selanjutnya, reaksi oksidasi isosafrol menjadi piperonal, pereaksi Na₂Cr₂O₇/H₂SO₄/H₂O/asam sulfanilat memberikan persentase hasil yang tertinggi yakni 80%, sedangkan dengan pereaksi KOH/DMSO – nitrobenzena, tidak berhasil. Untuk tahap terakhir yakni reaksi IGF piperonal menjadi sesamol, pereaksi MCPBA – CH₂Cl₂/KOH –

CH₃OH memberikan hasil sebesar 50%, sedangkan pereaksi H₂O₂/NaOH, tidak memberikan hasil. Selanjutnya, persentase hasil total transformasi safrol menjadi sesamol adalah 39%.

SUMMARY

The Effect of Reagent Type on the Percentage Yield of Transformation of Safrole [Isolated Product of Oil of Lawang (*Cinnamomum cullilawan*, BL)] into Sesamol

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In recent times, sesamol is widely used as antioxidant. Besides, it is also extensively utilized as the starting material in drug preparation, such as ayapin. Sesamol is naturally occurring in Sesame oil.

The presence of methylenedioxy, phenyl and hydroxyl groups in sesamol molecule enables it to be synthesized from other naturally occurring starting material, i.e. safrole, a product of isolation from Oil of Lawang (*Cinnamomum cullilawan*, BL). The transformation process needs some step of reactions, namely isomerization, oxidation and substitution.

For that reason, a study has been done about the effect of reagent type on the percentage yield of transformation of safrole, that was obtained from Oil of Lawang (*Cinnamomum cullilawan*, BL)] into sesamol. The Oil of Lawang used in this experiment is brand X obtained from Ambon. This study aims to determine which reagent produces the highest percentage yield in the transformation safrole into sesamol.

The safrole was firstly separated from Oil of Lawang by fractional distillation under reduced pressure. Afterwards, transformation safrole into isosafrole was done using five different reagents, namely *t*-BuOK/DMSO, KOH/*n*-BuOH, KOH/CH₂OHCH₂OH, NaOH/CH₂OHCH₂OH, and KOH/C₂H₅OH. The transformation of isosafrole into piperonal was carried out using three different reagents, namely Na₂Cr₂O₇/H₂SO₄/H₂O, Na₂Cr₂O₇/H₂SO₄/H₂O/sulfanilic acid, and KOH/DMSO/nitrobenzene and the transformation of piperonal into sesamol was implemented using MCPBA-CH₂Cl₂/KOH-CH₃OH and H₂O₂/NaOH reagents.

The separation of synthesis products were conducted with distillation under reduced pressure and column chromatography. The purities of all products were tested with thin layer chromatography and determination of the physical constants, such as refractive index, melting point, and density. Thereafter the structures were identified with infrared spectrophotometry, ¹H and ¹³C nuclear magnetic resonance spectrometry, and mass spectrometry.

The experimental result showed that the percentage yield of safrole obtained from Oil of Lawang was 7.05%. Isomerization product of safrole into isosafrole with the highest percentage yield (97%) was given by *t*-BuOK/DMSO. The oxidation of isosafrole into piperonal by means of Na₂Cr₂O₇/H₂SO₄/H₂O/sulfanilic acid gave the highest percentage yield, i.e. 80%, while KOH/DMSO – nitrobenzene reagent failed to produce piperonal. For the last step, the reagent MCPBA-CH₂Cl₂/KOH-CH₃OH gave 50% yield of sesamol, while H₂O₂/NaOH reagent gave no product at all. The total percentage yield of transformation of safrole into sesamol was therefore 39%.

ABSTRACT

The Effect of Reagent Type on the Percentage Yield of Transformation of Safrole [Isolated Product of Oil of Lawang (*Cinnamomum cullilawan*, BL)] into Sesamol

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A study has been carried out to find out the effect of reagent type on the percentage yield of transformation of safrole [isolated from Oil of Lawang (*Cinnamomum cullilawan*, BL)] into sesamol. Three reactions were applied in the transformation process, namely isomerization, oxidation, and substitution. Five different reagents were used in isomerization, three different reagents in oxidation, and two different reagents in substitution. This study aims to determine which reagent produces the highest percentage yield in the transformation safrole into sesamol.

Separation of the synthesis products was conducted by distillation under reduced pressure and column chromatography. The purities of all products were tested with thin layer chromatography and determination of physical constants, such as refractive index, melting point, and density, while the structures were identified by infrared spectrophotometry, ^1H and ^{13}C nuclear magnetic resonance spectrometry and mass spectrometry.

The experimental result showed that the content of safrole in Oil of Lawang was 7.05%. In safrole isomerization into isosafrole, the highest percentage yield (97%) was given by *t*-BuOK/DMSO. In oxidation of isosafrole into piperonal, the highest percentage yield (80%) was given by $\text{Na}_2\text{Cr}_2\text{O}_7/\text{H}_2\text{SO}_4/\text{H}_2\text{O}/\text{sulfanilic acid}$. The highest percentage yield of piperonal substitution into sesamol (50%) was given by MCPBA- $\text{CH}_2\text{Cl}_2/\text{KOH}-\text{CH}_3\text{OH}$ reagent. Therefore, the total percentage yield of safrole transformation into sesamol is 39%.

Key words: *Cinnamomum cullilawan*, BL; Safrole, Isosafrole, Piperonal, Sesamol