# SINTESIS 2',4'-DIMETIL-3,4-METILENDIOKSIKALKON DARI PIPERONAL DAN 2,4-DIMETILASETOFENON MENGGUNAKAN KATALIS NaOH DAN UJI ANTIBAKTERI TERHADAP *S. aureus* DAN *E. coli*


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**J2C003117**

## RINGKASAN

Kalkon merupakan senyawa flavonoid yang memiliki aktivitas biologis sebagai antibakteri, akan tetapi ketersediaannya di alam sangat terbatas. Penelitian sebelumnya melaporkan bahwa kalkon dapat disintesis melalui reaksi Claisen-Schmidt. Literatur lain (Alam, 2004) menyebutkan bahwa flavonoid dengan gugus metilendioksi (-O-$CH_2$-O-) memiliki aktivitas sebagai antibakteri. Penelitian ini bertujuan untuk memperoleh senyawa 2',4'-dimetil-3,4-metilendioksikalkon dari 2,4-dimetilasetofenon dan piperonal melalui reaksi kondensasi Claisen-Schmidt dengan menggunakan katalis NaOH, mengetahui pengaruh konsentrasi katalis NaOH dalam reaksi kondensasi Claisen-Schmidt dan mengetahui aktivitasnya sebagai antibakteri terhadap *S. aureus* (gram positif) dan *E. coli* (gram negatif).

Tahap awal adalah sintesis 2,4-dimetilasetofenon melalui reaksi Asilasi Friedel-Craft dengan menggunakan m-xylena dan anhidrida asam asetat sebagai bahan awal, $CH_2Cl_2$ sebagai pelarut dan $AlCl_3$ sebagai katalis. Reaksi ini dilakukan selama 45 menit pada suhu 50$^oC$. Campuran selanjutnya dituangkan ke dalam campuran es akuades dan HCl pekat. Produk diekstraksi menggunakan eter kemudian NaOH 10%, larutan dikeringkan dengan $MgSO_4$ anhidrat dan pelarutnya diuapkan dengan *rotary evaporator*. Produk dipisahkan dengan distilasi fraksinasi kemudian dianalisis dengan GC-MS. Tahap selanjutnya adalah sintesis 2',4'-dimetil-3,4-metilendioksikalkon melalui reaksi Claisen-Schmidt menggunakan piperonal dan 2,4-dimetilasetofenon sebagai bahan awal, etanol sebagai pelarut dan NaOH sebagai katalis. Reaksi dilakukan selama 3 jam pada suhu kamar. Produk diekstraksi menggunakan $CH_2Cl_2$ dan pelarut dievaporasi menggunakan *rotary evaporator*. Optimasi sintesis dilakukan dengan variasi konsentrasi NaOH (%b/v) 40%, 50%, 60%, 70% dan 80%. Produk ditentukan titik lelehnya, diidentifikasi dengan KLT kemudian dielusidasi dengan UV-Vis dan FT-IR. Aktivitas antibakteri produk diuji terhadap *S. aureus* dan *E. coli* dengan metode difusi cakram kertas.

Senyawa 2,4-dimetilasetofenon hasil sintesis berupa cairan coklat dengan berat jenis 0,961 g/ml dan kelimpahan sebesar 55,61%. Sedangkan senyawa 2',4'-dimetil-3,4-metilendioksikalkon hasil sintesis berupa padatan kuning dengan titik leleh 62-79$^oC$. Spektra UV-Vis menunjukkan 2 puncak karakteristik senyawa kalkon, yaitu $\lambda$=258 nm menunjukkan adanya sistem benzoil dan $\lambda$=348 nm menunjukkan adanya sistem sinamoil. Spektra FT-IR menunjukkan serapan gugus karbonil (C=O) pada bilangan gelombang 1647,1 $cm^{-1}$; serapan C=C aromatik ditunjukkan pada bilangan gelombang 1500,5 $cm^{-1}$; serapan C=C alkena ditunjukkan pada bilangan gelombang 1604,7 $cm^{-1}$, serapan C-O dari gugus -O-$CH_2$-O- muncul pada 1242,1 $cm^{-1}$, serapan C-H dari gugus -$CH_2$- muncul pada 1446,0 $cm^{-1}$ dan serapan pada bilangan gelombang 2920 $cm^{-1}$ menunjukkan vibrasi $C_{sp3}$-H dari gugus metil ($CH_3$). Data tersebut menunjukan bahwa 2',4'-dimetil-3,4-metilendioksikalkon telah terbentuk. Produk optimum diperoleh pada konsentrasi NaOH 60% dengan rendemen sebesar 53,599%. Hasil uji antibakteri menunjukkan bahwa 2',4'-dimetil-3,4-metilendioksikalkon memiliki aktivitas sebagai antibakteri terhadap *S. aureus* (gram positif) dan *E. coli* (gram negatif).

## SUMMARY

Chalcone is a flavonoid compound that has antibacterial activity which is only found on nature in a limited number. Previous research reported that chalchone could be synthesized through Claisen-Schmidt. Furthermore, Alam (2004) demonstrated that flavonoid containing methylenedioxy group (-O-$CH_2$-O-) had antibacterial activity. The objective of this study were to synthesyze 2',4'-dimethyl-3,4-methylenedioxychalcone from 2,4-dimethylacetophenone and piperonal through Claisen-Schmidt reaction using NaOH as catalyst, to find out the influence of NaOH concentration on Claisen-Schmidt reaction, and to find out its antibacterial activity against *S. aureus* ($G^+$) and *E. Coli* ($G^-$).

The first step was the synthesis of 2,4-dimethylacetophenone through Friedel-Craft Acylation by using m-xylene and acetic anhydride as the starting materials, $CH_2Cl_2$ as the solvent and $AlCl_3$ as the reaction catalyst. This reaction was carried out at 50°C for 45 minutes. The mixture was then poured into a mixture of chipped ice and concentrated HCl. The product was extracted using ether and then NaOH 10%, the extracted product was dried by using anhydrous $MgSO_4$ and the solvent was evaporated by rotary evaporator. The product was then isolated by fractional distillation method and analyzed using GC-MS. The sebsequent step in this study was the synthesis of 2',4'-dimethyl-3,4-methylendioxychalcone through Claisen-Schmidt reaction by using piperonal and 2,4-dimethylacetophenon as the starting materials, ethanol as the solvent and NaOH as the reaction catalyst. The reaction was carried out at room temperature for 3 hours. The product was extracted using $CH_2Cl_2$ and the solvent was then evaporated by rotary evaporator. Synthesis optimization was done by varying NaOH concentration (%w/v) to 40%, 50%, 60%, 70% and 80%. The products was characterized for their melting point, identified by TLC method and analyzed by UV-Vis and FT-IR. Antibacterial activities of products were tested against two human pathogenic bacteria, *S. aureus* and *E. coli* by using filter paper disc diffusion method.

A brown liquid of 2,4-dimethylacetophenon with the density of 0.961 g/mL and abundance of 55.61% was synthesized in this research. Furthermore, a yellow solid of 2',4'-dimethyl-3,4-methylendioxychalcone with the melting point of 62-79 °C was also synthesized. The UV-Vis spectra showed the presence of two characteristic peaks at 258 nm and 348 nm which represented the absorption of benzoil and cynamoil systems respectively. Meanwhile, the FTIR spectra showed the absorption of carbonyl (C=O) at 1647.1 $cm^{-1}$, absorption of aromatic C=C groups at 1500.5 $cm^{-1}$, absorption of C=C alkene at 1604.7 $cm^{-1}$, absorption of -C-O- of methylenedioxy (-O-$CH_2$-O-) at 1242.1 $cm^{-1}$, absorption of $C_{sp3}$-H of methylene (-$CH_2$-) at 1446.0 $cm^{-1}$ and the vibration of $C_{sp3}$-H of methyl group ($CH_3$) at 2920.0 $cm^{-1}$. From the mentioned data, it could be concluded that 2',4'-dimethyl-3,4-methylenedioxychalcone had been successfully synthesized. The optimum product was obtained on the NaOH concentration of 60% with the rendement of 53.599%. Furthermore, based on its antibacterial test, it could be concluded that 2',4'-dimethyl-3,4-methylenedioxychalcone could also play a role as antibacteria against *S. aureus* ($G^+$) and *E. coli* ($G^-$).

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