

# **SINTESIS ASAM C-ARILKALIKS[4]PIROGALOLARENA SULFONAT DAN UJI AKTIVITASNYA SEBAGAI ORGANOKATALIS PADA SINTESIS BIODIESEL BERBAHAN DASAR ASAM PALMITAT**

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## **INTISARI**

Sintesis asam C-arilkaliks[4]pirogalolarena sulfonat dan uji aktivitasnya sebagai organokatalis pada sintesis biodiesel berbahan dasar asam palmitat telah dilakukan. Penelitian ini bertujuan untuk mensintesis senyawa 4-etoksi-3-metoksibenzaldehida, C-arilkaliks[4]pirogalolarena, asam C-arilkaliks[4]pirogalolarena sulfonat dan uji aktivitasnya sebagai organokatalis pada sintesis biodiesel berbahan dasar asam palmitat.

Penelitian ini diawali dengan sintesis 4-etoksi-3-metoksibenzaldehida melalui reaksi alkilasi vanilin dengan dietilsulfat dan katalis NaOH. Sintesis asam C-arilkaliks[4]pirogalolarena sulfonat dilakukan dalam 2 tahap reaksi. Tahap pertama adalah reaksi kondensasi antara pirogalol dengan variasi aldehida aromatik antara lain 4-etoksi-3-metoksibenzaldehida, vanilin dan benzaldehida dalam pelarut etanol dan katalis HCl menghasilkan C-4-etoksi-3-metoksifenilkaliks[4]pirogalolarena (PgPh4OEt3OMe), C-4-hidroksi-3-metoksifenilkaliks[4]pirogalolarena (PgPh4OH3OMe) dan C-fenilkaliks[4]pirogalolarena (PgPh) dilanjutkan reaksi sulfonasi dengan asam sulfat pekat menghasilkan asam C-4-etoksi-3-metoksifenilkaliks[4]pirogalolarena sulfonat (PgPh4OEt3OMeS), asam C-4-hidroksi-3-metoksifenilkaliks[4]pirogalolarena sulfonat (PgPh4OH3OMeS) dan asam C-fenilkaliks[4]pirogalolarena sulfonat (PgPhS). Senyawa hasil sintesis dikarakterisasi dengan FTIR, GC-MS, LC-MS, <sup>1</sup>H-NMR dan <sup>13</sup>C-NMR. Uji aktivitas sebagai organokatalis pada sintesis biodiesel dilakukan melalui reaksi esterifikasi asam palmitat dan metanol berdasarkan variasi waktu reaksi, mol% katalis dan suhu reaksi. Aktivitas sebagai organokatalis dibandingkan dengan asam sulfat pekat sebagai kontrol positif.

Hasil penelitian menunjukkan bahwa 4-etoksi-3-metoksibenzaldehida, PgPh4OEt3OMe, PgPh4OH3OMe, PgPh, PgPh4OEt3OMeS, PgPh4OH3OMeS dan PgPhS telah berhasil disintesis dengan persen hasil berturut-turut yaitu 73,0; 98,8; 93,3; 99,7; 85,3; 88,9 dan 82,5%. PgPh4OH3OMeS menunjukkan aktivitas terbaik sebagai organokatalis pada sintesis biodiesel dari asam palmitat dan metanol dengan persen hasil metil palmitat sebesar 91,9% pada waktu reaksi 4 jam, 4 mol% katalis dan suhu reaksi 65 °C.

Kata kunci : 4-etoksi-3-metoksibenzaldehida, kaliks[4]pirogalolarena, sulfonasi, organokatalis, biodiesel

# **SYNTHESIS OF C-ARYLCALIX[4]PYROGALLOLARENE SULFONIC ACID AND THEIR ACTIVITY ASSAY AS ORGANOCATALYST IN THE SYNTHESIS OF BIODIESEL BASED ON PALMITIC ACID**

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## ***ABSTRACT***

Synthesis of C-arylcax[4]pyrogallolarene sulfonic acid and their activity assay as organocatalyst in the synthesis of biodiesel based on palmitic acid have been conducted. The aims of this research are to synthesize 4-ethoxy-3-methoxybenzaldehyde, C-arylcax[4]pyrogallolarene, C-arylcax[4]pyrogallolarene sulfonic acid and their activity assay as organocatalyst in the synthesis of biodiesel based on palmitic acid.

This research was initiated by synthesizing 4-ethoxy-3-methoxybenzaldehyde by alkylation of vanillin with diethylsulfate and NaOH catalyst. Synthesis of C-arylcax[4]pyrogallolarene sulfonic acid was carried out in two steps of reaction. The first step was condensation reaction between pyrogallol with variations of aromatic aldehyde i.e. 4-ethoxy-3-methoxybenzaldehyde, vanillin and benzaldehyde in ethanol solvent and HCl catalyst to produce C-4-ethoxy-3-methoxyphenylcalix[4]pyrogallolarene (PgPh4OEt3OMe), C-4-hydroxy-3-methoxyphenylcalix[4]pyrogallolarene (PgPh4OH3OMe) and C-phenylcalix[4]pyrogallolarene (PgPh) followed by sulphonation reaction with neat sulphuric acid to produce C-4-ethoxy-3-methoxyphenylcalix[4]pyrogallolarene sulfonic acid (PgPh4OEt3OMeS), C-4-hydroxy-3-methoxyphenylcalix[4]pyrogallolarene sulfonic acid (PgPh4OH3OMeS) and C-phenylcalix[4]pyrogallolarene sulfonic acid (PgPhS). The synthesized compounds were characterized by FTIR, GC-MS, LC-MS, <sup>1</sup>H-NMR and <sup>13</sup>C-NMR. Activity assay as organocatalyst in the synthesis of biodiesel was carried out through esterification of palmitic acid and methanol based on variation of reaction time, mol% catalyst and reaction temperature. Activity as organocatalyst was compared with sulphuric acid as positive control.

The results showed that 4-ethoxy-3-methoxybenzaldehyde, PgPh4OEt3OMe, PgPh4OH3OMe, PgPh, PgPh4OEt3OMeS, PgPh4OH3OMeS and PgPhS have been successfully synthesized with percent yield of 73.0; 98.8; 93.3; 99.7; 85.3; 88.9 and 82.5% approximately. PgPh4OH3OMeS showed the best activity as an organocatalyst in the synthesis of biodiesel from palmitic acid and methanol with methyl palmitate percent yield which was 91.9% at the reaction time of 4 hours, 4 mol% of catalyst and reaction temperature of 65 °C.

Keywords : 4-ethoxy-3-methoxybenzaldehyde, calix[4]pyrogallolarene, sulphonation, organocatalyst, biodiesel