SINTESIS SENYAWA TABIR SURYA DAN ANTIOKSIDAN TURUNAN KALIKS[4]RESORSINARENA SERI BENZOIL DAN SINAMOIL DARI SALISILALDEHIDA
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Sintesis Senyawa Tabir Surya dan Antioksidan Turunan Kaliks[4]Resorsinarena Seri Benzoil dan Sinamoil dari Salisilaldehida

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Intisari

Telah dilakukan sintesis senyawa baru seri benzoil-sinamoil fenilkaliks[4]resorsinarena dan benzoat-sinamat fenilkaliks[4]resorsinarena. Sintesis dilakukan dari salisilaldehida dan resorsinol melalui reaksi alkilasi, substitusi elektofilik aromatik-siklisasi, reaksi esterifikasi, reaksi asilasi Friedel-Craft dengan $AlCl_3$, dan reaksi hidrolisis. Elusidasi struktur dilakukan dengan spektrofotometer FTIR dan NMR, GC-MS,dan LC-MS. Sintesis senyawa turunan benzoil-sinamoil fenilkaliks[4]resorsinarena yang diuji aktifitas tabir surya dan antioksidan terdiri atas senyawa benzoil C-2-hidroksifenilkaliks[4]resorsinarena (BCHFKR atau senyawa **13**), benzoil C-2-metoksifenilkaliks[4]resorsinarena (BCMFKR/**14**), benzoil C-2-etoksi-fenilkaliks[4]resorsinarena (BCEFKR/**15**), sinamoil C-2-hidroksifenilkaliks[4]-resorsinarena (SCHFKR/**16**), sinamoil C-2-metoksifenilkaliks[4]resorsinarena (BCMFKR/**17**), sinamoil C-2-etoksifenilkaliks[4]resorsinarena (BCEFKR/**18**). Seri benzoat-sinamat fenilkaliks[4]resorsinarena hanya diuji aktifitas sebagai tabir surya terdiri atas 2-hidroksifenilkaliks[4]resorsinaril oktabenzoat (CHFKROB/**19**), C-2-metoksifenilkaliks[4]resorsinaril oktabenzoat (CMFKROB/**20**), C-2-etoksifenil-kaliks[4]resorsinaril oktabenzoat (CEFKROB/**21**), C-2-hidroksifenilkaliks[4]-resorsinaril oktasinamat, (CHFKROS/**22**), C-2-metoksifenilkaliks[4]resorsinaril oktasinamat (CMFKROS/**23**), dan 2-etoksifenilkaliks[4]resorsinaril oktasinamat (CEFKROS/**24**). Senyawa awal fenilkaliks[4]resorsinarena yang diuji sebagai antioksidan antara lain C-2-hidroksifenilkaliks[4]resorsinarena (CHFKR/**1**), C-2-metoksifenilkaliks[4]resorsinarena (CMFKR/**2**) dan C-2-etoksifenilkaliks[4]-resorsinarena (CEFKR/**3**). Pengujian aktifitas tabir surya dilakukan dengan metode *in vitro* menggunakan spektrofotometer dan MTT *assay*, sedangkan uji antioksidan dilakukan dengan DPPH *assay*.

Rendemen reaksi senyawa fenilkaliks[4]resorsinarena yang diperoleh berturut-turut : 93,14 % (**1**); 96,93 % (**2**) dan 99,26 % (**3**). Rendemen reaksi seri benzoil-sinamoil fenilkaliks[4]resorsinarena berturut-turut : 88,65 % (**13**); 88,65 % (**14**); 74,56% (**15**); 44,27 % (**16**); 67,81 % (**17**) dan 79,26% (**18**). Rendemen reaksi seri benzoat-sinamat fenilkaliks[4]resorsinarena berturut-turut: 67,06% (**19**); 81,60% (**20**); 74,32% (**21**); 63,12% (**22**); 80,87% (**23**) dan 54,20% (**24**).

Uji aktifitas tabir surya dengan menggunakan spektrofotometer UV-Vis menghasilkan nilai *sun protection factor* (SPF) tinggi berturut-turut 162,93 (CHFKROS/20); 57,41 (CMFKROS/22); 30,20 (SCEFKR/18) dan 12,08

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(CEFKROS/24) pada konsentrasi 20 ppm. Senyawa dengan tabir surya tinggi memiliki nilai EC_{50} berturut-turut 0,88 ppm (CHFKROS/20); 1,23 ppm (CMFKROB/21) dan 1,47 ppm (SCEFKR/18). Sebagai pembanding nilai EC_{50} Sunzone dan Parasol masing-masing 1,10 dan 0,88 ppm. Nilai IC_{50} senyawa terhadap sel Vero berturut-turut adalah : 6486,60 ppm (BCMFKR/13); 3800,89 ppm (CEFKROS/24); 2626,96 ppm (CEFKROB/23); 1949,40 ppm (CMFKROB/21); 1823,27 ppm (SCHFKR/15) dan 1522,79 ppm (CMFKROS/22). Data tersebut menunjukkan bahwa BCMFKR/13, CEFKROS/24, CEFKROB/23, CMFKROB/21, SCHFKR/15 dan CMFKROS/22 bersifat relatif tidak toksik terhadap sel Vero. Uji aktifitas antioksidan dengan DPPH diperoleh senyawa yang memiliki sifat antioksidan kuat yaitu CMFKR, CHFKR, dan CEFKR dengan nilai ES_{50} berturut-turut : 43,45; 49,88 dan 54,58 µg/mL. Senyawa BCMFKR/13 memiliki aktivitas antioksidan sedang dengan nilai ES_{50} 123,87 µg/mL dan senyawa yang mempunyai aktivitas antioksidan yang lemah beserta nilai ES_{50} antara lain: SCMFKR/17 (151,64 µg/mL); BCHFKR/13 (198,29 µg/mL); BCEFKR/15 (642,37 µg/mL) dan SCEFKR/18 (740,08 µg/mL). Sebagai pembanding nilai ES_{50} BHT sebesar 21,67 µg/mL.

Kata Kunci : sintesis, tabir surya, antioksidan, turunan kaliks[4]resorsinarena, benzoil, sinamoil dan salisilaldehida.

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Synthesis of Sunscreen and Antioxidant Compound of Calix[4]Resorcinarene Derivatives of Benzoyl-Cinnamoyl Series from Salicylaldehyde

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Abstract

Synthesis of benzoate-cinnamate and benzoyl-cinnamoyl phenylcalix[4]-resorcinarene series has been conducted. The synthesis was carried out from salicylaldehyde and recorcinol via alkylation, aromatic electrophilic substitution-cyclization, esterification, Friedel-Craft acylation reaction with $AlCl_3$ catalyst, and hydrolysis reaction. Structural elucidation of product was performed using FTIR and NMR spectrophotometer, GC-MS and LC-MS. The benzoyl-cinnamoyl calix[4]resorcinarene series compound consist of the benzoyl C-2-hidroxyphenyl-calix[4]resorcinarene (BCHFKR/**13** compound), benzoil C-2-methoxyphenylcalix[4]-resorcinarene (BCMFKR/**14**), benzoyl C-2-ethoxyphenylcalix[4]resorcinarene (BCEFKR/**15**), cinnamoyl C-2-hidroxyphenylcalix[4]resorcinarene (SCHFKR/**16**), sinamoil C-2-methoxyphenylcalix[4]resorsinarene (BCMFKR/**17**), cinnamoyl C-2-ethoxyphenyl-calix[4]resorcinarene (BCEFKR/**18**) were assay activity of sunscreen and antioxidant. The benzoate-cinnamate phenylcalix[4]resorcinarene series were examined as sunscreen consist of C-2-hidroxyphenylcalix[4]resorcinaryl octabenzoate (CHFKROB/**19**), C-2-hidroxyphenylcalix[4]resorcinaryl octacinnamate, (CHFKROS/**20**), C-2-methoxyphenylcalix[4]resorcinaryl octabenzoate (CMFKROB/**21**), C-2-methoxy-phenylcalix[4]resorcinaryl octacinnamate (CMFKROS/**22**), C-2-ethoxy-phenylcalix[4]resorcinaryl octabenzoate (CEFKROB/**23**), and 2-ethoxyphenyl-calix[4]resorcinaryl octacinnamate (CEFKROS/**24**). The initial phenylcalix[4]-recorcinarene was examined antioxidant activity consist of C-2-hidroxyphenylcalix[4]-resorcinarene (CHFKR/**1**), C-2-methoxyphenylcalix[4]resorcinarene (CMFKR/**2**), and C-2-ethoxy-phenylcalix[4]resorcinarene (CEFKR/**3**). The examination of sunscreen activity was done by in vitro method using UV-Vis spectrophotometer and MTT assay, while that of antioxidant assay was done using DPPH methods.

The percent yields the reaction of the phenylcalix[4]resorcinarene series were 93.14% (**1**); 96.93% (**2**) and 99.26% (**3**) respectively. Whereas percent yields of benzoyl-cinnamoyl phenylcalix[4]resorcinarene series were 88.65% (**13**); 88.65% (**14**); 74.56% (**15**); 44.27% (**16**); 67.81% (**17**) and 79.26% (**18**). The yield of benzoate-cinnamate phenylcalix[4]resorcinarene series were: 67.06% (**19**); 81.60% (**22**); 74.32% (**20**); 63.12% (**23**); 80.87% (**21**) and 54.20% (**24**) respectively.

The result of activity test as suncreen using UV-Vis spectrophotometer were SPF (sun protection factor) enough high value of 162.93 (CHFKROS/20); 57.41

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(CMFKROS/22); 30.20 (SCEFKR/18) and 12.08 (CEFKROS/24) at concentration 20 ppm respectively. The compounds have high activity as suncreen using MTT assay with effective concentration $_{50}$ (EC_{50}) values of 0.88 ppm (CHFKROS/20); 1.23 ppm (CMFKROB/21) and 1.47 ppm (SCEFKR/18) respectively. Whereas the EC_{50} value of Sunzone and Parasol are 1.1 and 0.88 ppm. The compound have the high inhibitor concentration $_{50}$ (IC_{50}) ie : 6486.60 ppm (BCMFKR/13); 3800.89 ppm (CEFKROS/24); 2626.96 ppm (CEFKROB/23); 1949.40 ppm (CMFKROB/21); 1823.27 ppm (SCHFKR/16) and 1522.79 ppm (CMFKROS/22). The compound of BCMFKR/13, CEFKROS/24, CEFKROB/23 and CMFKROB/21 shown relatively not toxic toward Vero cell. The result antioxidant activity of the compound with high activity are CMFKR/2, CHFKR/1 and CEFKR/3 with the effective scavenging $_{50}$ (ES_{50}) value 43.45; 49.88 and 54.58 µg/mL respectively. The BCMFKR/13 has enough activity with ES_{50} 123.87 µg/mL. Another compound of SCMFKR/17, BCHFKR/13, BCEFKR/15 and SCEFKR/18 have weak activity antioxidant with ES_{50} value 151.64; 198.2; 642.37 and 740.08 µg/mL respectively. Whereas the ES_{50} value of BHT is 21.67 µg/mL.

Keyword: synthesis, sunscreen, antioxidant, calix[4]resorcinarene and salicyaldehyde.