



SELF-ASSEMBLY OF ANTHRANILAMIDE DERIVATIVES AS ORGANOGELS

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ABSTRACT

A ring-opening reaction of isatoic anhydride with alkylamines to form benzamides followed by an acetylation reaction was carried out to synthesise acetylated benzamides. The self-assembling properties of the acetylated benzamides were tested in 25 solvent systems. Their structural and mechanical characteristics were analysed through concentration-dependent spectroscopy and rheology, respectively.

The synthesis of benzamides was done by adding an *n*-alkylamine, i.e. decylamine, dodecylamine, or tetradecylamine, to isatoic anhydride in water at room temperature for around 8 h to produce 2-amino-*N*-decylbenzamide (**3a**), 2-amino-*N*-dodecylbenzamide (**3b**), and 2-amino-*N*-tetradecylbenzamide (**3c**). A subsequent acetylation reaction by adding acetic anhydride and triethylamine to **3a-c** in DCM under inert Ar atmosphere for 12 h produced the acetylated benzamides: 2-acetamido-*N*-decylbenzamide (**4a**), 2-acetamido-*N*-dodecylbenzamide (**4b**), and 2-acetamido-*N*-tetradecylbenzamide (**4c**). Structure elucidation was done by ¹H-NMR, ¹³C-NMR, and FT-IR spectroscopies. After the self-assembly tests, their structural characteristics were analysed through concentration-dependent ¹H-NMR and UV-Vis spectroscopies and for the best gels formed in the self-assembly test, their mechanical properties were tested through rheology in the frequency and strain sweep tests.

From the research done, it was found that the synthesis of **3a**, **3b**, and **3c** had been successfully done with yields 92, 94, and 97%, respectively. The acetylation of **3a-c** to form **4a**, **4b**, and **4c** had also been successfully done with yields of 77, 89, and 95%, respectively. Through the analysis, it was found that **4a** in 30% DMSO was the most stable gel though not as stiff as the rest.

Keywords: anthranilamide, isatoic anhydride, organogels, self-assembly



PERAKITAN MANDIRI SENYAWA TURUNAN ANTRANILAMIDA SEBAGAI ORGANOGEL

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INTISARI

Reaksi pembukaan cincin isatoik anhidrida dengan alkilamina untuk mensintesis benzamida yang selanjutnya diasetilasi untuk menghasilkan benzamida terasetilasi telah dilakukan. Sifat-sifat *self-assembly*, atau perakitan mandiri, dari benzamida terasetilasi diuji dalam 25 sistem pelarut berbeda. Struktur dan sifat mekanik juga dianalisa melalui spektroskopi dan reologi.

Sintesis benzamida dilakukan dengan menambahkan *n*-alkilamina, yaitu desilamina, dodesilamina, dan tetradesilamina, ke dalam larutan isatoik anhidrida dalam air pada suhu ruang selama 8 jam untuk menghasilkan 2-amino-*N*-desilbenzamida (**3a**), 2-amino-*N*-dodesilbenzamida (**3b**), and 2-amino-*N*-tetradesilbenzamida (**3c**). Selanjutnya, reaksi asetilasi dilakukan dengan menambahkan anhidrida asetat dan trietilamina ke dalam campuran **3a-c** dalam diklorometana dan sistem atmosfer gas argon selama 12 jam menghasilkan benzamida terasetilasi: 2-asetamido-*N*-desilbenzamida (**4a**), 2-asetamido-*N*-dodesilbenzamida (**4b**), and 2-asetamido-*N*-tetradesilbenzamida (**4c**). Elusidasi struktur dilakukan dengan spektroskopi ¹H-NMR, ¹³C-NMR, dan FTIR. pengujian sifat *self-assembly*, struktur dianalisis melalui spektroskopi NMR dan UV-Vis dan sifat mekanik ditentukan menggunakan uji reologi yaitu uji frekuensi dan ketegangan.

Hasil percobaan menunjukkan bahwa senyawa **3a**, **3b**, dan **3c** telah berhasil disintesis dengan rendemen berturut-turut sebesar 92, 94, dan 97%. Reaksi asetilasi terhadap senyawa **3a-c** juga berhasil dilakukan dan diperoleh produk **4a**, **4b**, dan **4c** dengan rendemen berturut-turut sebesar 77, 89, dan 95%. Hasil analisis membuktikan bahwa senyawa **4a** dalam 30% DMSO merupakan gel yang paling stabil meskipun sifat kekakuannya tidak menunjukkan hasil terbaik.

Kata kunci: anthranilamida, isatoik anhidrida, *organogels*, *self-assembly*