Preparation of Laurel Oil Alkanolamide from Laurel Oil

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ABSTRACT: A low-temperature synthesis of laurel oil alkanolamides directly from laurel oil and ethanolamine was carried out in essentially quantitative yields. The ethanolamine/laurel oil molar ratio used was 10:1. Even though amine served as a catalyst in the reaction, we used sodium methoxide at a ratio of 0.2–2% as a second catalyst. The reaction was complete in 1–9 h at room temperature. The identity of the amide was confirmed by IR and ^{13}C NMR spectroscopy.

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KEY WORDS: Alkanolamides, amidation, ethanolamine, laurel oil, laurel oil alkanolamide.

Laurel oil is obtained by either direct extraction or boiling-water treatment from the black olive-sized fruits of *Laurus nobilis* L., which is found in the Mediterranean region. Approximately 45% of the oil is lauric acid, with minor amounts of oleic, linoleic, and palmitic acids (1).

Fatty alkanolamides have a broad spectrum of uses, e.g., in shampoos, detergents, cosmetics, lubricants, foam control agents, and water repellents (2). These are produced industrially from FA or fatty methyl esters and alkanolamines, such as ethanolamine, by heating at 140–160°C for 6–12 h (3).

Recently, Feairheller and coworkers (3) determined that secondary fatty amides may be prepared in high yield and purity by reaction of TG, such as tallow or vegetable oils, with primary amines at room temperature. The amine was used in 10:1 molar excess and served as reagent, as solvent for the fat and the fatty amide that formed, and possibly as a basic catalyst for the reaction.

An alkanolamide was synthesized from laurel oil by employing this method; the reaction was completed in 9 h. Although this reaction was catalyzed by the amine, a stronger base may shorten the reaction time. It is known from previous work that sodium methoxide can be used as a catalyst for the synthesis of alkanolamides (4).

EXPERIMENTAL PROCEDURES

Materials. Ethanolamine was purchased from Aldrich Chemical Co. (Milwaukee, WI) and was used without further purification. The laurel oil was obtained by boiling-water treatment (5) of the fruits from plants grown in Edirne city province.

Instrumentation. Instruments used in this investigation were a Shimadzu IR spectrophotometer (Model 470), Varian nuclear magnetic resonance spectrometer (Model Mercury Plus 300), Agilent Technologies 6890 N Network gas chromatography system, and Gallenkamp m.p. apparatus.

Extraction of laurel oil. Laurel fruits, which mature in autumn, were picked from trees, washed with water, and dried at room temperature. Eighty-five grams of fruits were broken into small pieces and transferred to a 1000-mL beaker. Water (500 mL) was added, and the mixture was boiled for 5 h. The oil was skimmed from the top of the water during the boiling period by spatula. The oil was dissolved in 100 mL of hexane, dried over anhydrous sodium sulfate, and filtered. The hexane was rotary-evaporated, yielding 6.4 g (7.5%) of yellowish oil.

IR: 3472, 2928, 1740, 1462, 1379, 1241, 1161, 732 cm^{-1}. ^{13}C NMR(CDCl$_3$): δ, 14.29, 22.87, 25.06, 27.37, 27.42, 29.31, 29.37, 29.46, 29.52, 29.67, 29.72, 29.80, 29.85, 29.89, 29.96, 32.10, 34.25, 62.31, 130.21, 173.51. GC [FA, % w/w (C_{number})]: 0.19 ($C_{8:0}$), 0.67 ($C_{10:0}$), 28.75 ($C_{12:0}$), 1.73 ($C_{14:0}$), 25.24 ($C_{16:0}$), 0.57 ($C_{16:1}$), 1.79 ($C_{17:0}$), 1.36 ($C_{17:1}$), 3.17 ($C_{18:0}$), 29.19 ($C_{18:1}$), 2.9 ($C_{18:2}$), 3.7 ($C_{22:0}$), 0.2 ($C_{22:1}$), 0.3 ($C_{24:0}$).

General procedure for the synthesis of alkanolamide from laurel oil. Laurel oil (2.5 g, 0.003 mol), ethanolamine (1.83 g, 0.03 mol), and sodium methoxide (86.4 mg, 2% by weight) were weighed into a 50-mL round-bottomed flask and reacted with agitation at room temperature for 1 h. The reaction mixture was dissolved in 60 mL diethyl ether and placed in a separatory funnel. The ether phase was washed with 10 mL of 5% aqueous hydrochloric acid to remove excess amine and glycerol. The ether layer was separated, washed with two 5-mL portions of water, boiled with 0.5 g of decolorizing charcoal to remove the yellow color, and filtered. The organic phase was dried over magnesium sulfate and the ether was rotary-evaporated. A white waxy laurel oil alkanolamide, m.p. 57–66°C, was obtained (2.27 g, 91% yield).

IR: 3424, 2928, 2096, 1638, 1561, 1488, 1459, 1328, 1062 cm^{-1}. ^{13}C NMR (CDCl$_3$): δ, 14.31, 22.77, 22.88, 25.85, 25.94, 27.43, 29.34, 29.46, 29.49, 29.53, 29.69, 29.73, 29.82, 29.90, 29.98, 31.73, 32.11, 36.86, 42.73, 62.70, 128.12, 128.28, 129.94, 130.23, 130.45, 174.81.

RESULTS AND DISCUSSION

When the initial reaction of laurel oil with ethanolamine (no catalyst) was carried out at room temperature and atmospheric pressure, the reaction was completed in 9 h. Product

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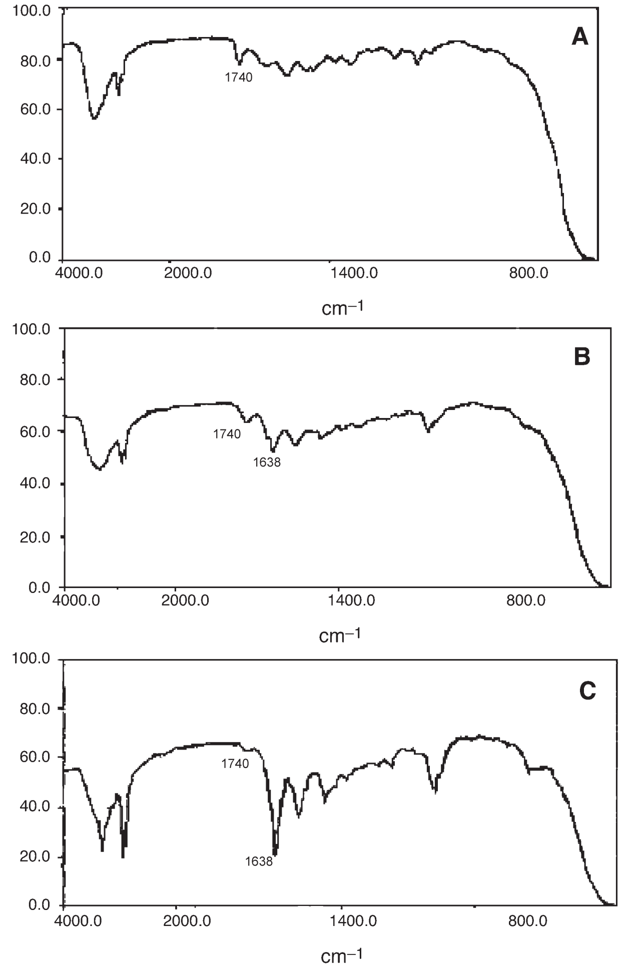

FIG. 1. IR spectra of aliquots at 1, 6, and 9 h of reaction.

formation and reactant disappearance were monitored by IR spectroscopy. Small samples (1–2 drops into diethyl ether) were removed hourly from the reaction mixtures, and IR absorption spectra were measured. Figure 1A is the spectrum of a sample taken from the reaction mixture after 1 h. The ester peak is at 1740 cm^{-1} . After reacting 6 h, the ester peak started to decrease while the amide peak at 1638 cm^{-1} started to increase (Fig. 1B). The ester peak almost disappeared after 9 h (Fig. 1C).

The concentration of sodium methoxide was determined to ascertain whether it had any effect on the rate of the reaction. Whereas 0.2% sodium methoxide (percent weight with respect to the total amount of laurel oil and ethanolamine) does not speed up the reaction, 0.75% sodium methoxide reduces the reaction time to 7 h from 9 h, and 1 or 2% sodium methoxide decreases the reaction time to approximately 1 h.

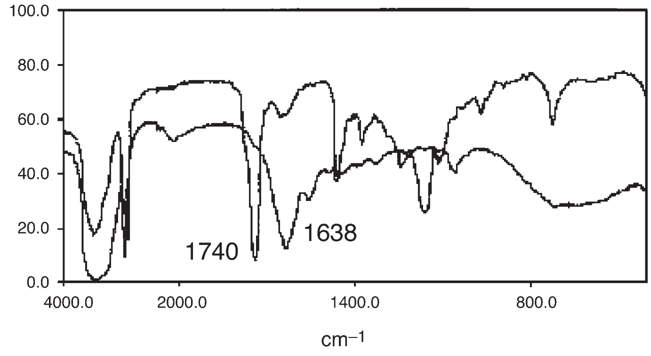

FIG. 2. IR spectra of the experiment with 2% CH_3ONa (the C=O peak of laurel oil is at about 1740 cm^{-1}, and the C=O peak of alkanolamide is about at 1638 cm^{-1}).

When 2% catalyst was used, the C=O peak (IR) of ester was completely converted to the C=O of amide (Fig. 2). In addition, the ^{13}C NMR spectrum of laurel oil alkanolamide gave the following characteristic peaks: 42.7 (CH_2NH), 62.7 (CH_2OH), and 174.8 ppm (CONH).

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