

## Experiment 8 = Production of Biodiesel =

**Objective<sup>1</sup>**      ➔ (OR CAN BE LABELED EQUIVALENTLY AS) “**INTRODUCTION**”

Experiment 8 investigates the production of biodiesel via base-catalyzed transesterification of long ester side chains (such as that found in household vegetable oil). The objective of the experiment is to obtain a full yield of biodiesel via this transesterification reaction and qualitatively analyze the resulting product through viscosity and flame tests. These direct observations will be made in reference to other known compounds. Lastly, a calculated percent yield will provide insight into the purity of the product formed.

**Methods (abbreviated)<sup>2</sup>**      ➔ (OR CAN BE LABELED EQUIVALENTLY AS) “**PROCEDURE**”

RXN (1) formation of sodium methoxide - Combined 0.2 g of sodium hydroxide (pellets) and 10 mL of methanol into a 100 mL round bottom flask and added a magnetic stir bar. Mixed the contents within the flask over a magnetic stirring plate until the entire solid sodium hydroxide dissolved, using a spatula as necessary to break up solid fragments.<sup>3</sup>

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<sup>1</sup> In the **Introduction**, provide succinct statements describing the chemical concept being examined, the purpose and/or expected outcomes of the experiment, and a short synopsis of the experimental protocol (any reactions or tests). Depending on the formality of the report, this section may also provide a direct illustration of the reaction scheme and list the chemical compounds with corresponding properties.

<sup>2</sup> In the **Methods**, each procedural statement should be completely objective. Use third person past tense. When making a procedural statement, be as *concise* and *precise* as possible: give all details necessary to replicate the experiment. Consider all conditions (time, reagents, temperature, quantity, etc.) at each step.

<sup>3</sup> Avoid using language that would animate any of the chemical compounds in consideration. (ex - In this sentence, you may equivalently say: “*The sodium hydroxide wanted to dissolve when put in the magnetic stirring plate but some stubborn solid fragments needed additional help via breaking up with a spatula.*” But this sentence references inanimate chemical compounds with (unnecessary) animated language!)

RXN (2) production of biodiesel - Combined 50 mL of vegetable oil to the 100 mL flask with the sodium methoxide solution. Fit the reaction vessel to a condenser. After the solution began to boil, ran a reflux through the reaction vessel.<sup>4</sup> After 10 minutes, placed the flask contents into a separatory funnel and allowed the solution to separate at room temperature for an additional 10 minutes. Then removed the lower glycerol layer into a 50 mL flask. The remaining top layer - the intended biodiesel product - was added to a pre-weighed 50 mL flask and the yield of biodiesel was recorded. A viscosity test and flame test were performed on samples of the biodiesel product with samples of known compounds (namely olive oil and methanol) to assess its purity and utility.<sup>5</sup>

## Observations<sup>6</sup>

Viscosity Test: *Upon direction inversion in a test-tube, biodiesel ran easier down the walls of the test tube than the olive oil (less viscous).*

Flame Test: *Upon subjecting a different cotton balls (soaked in biodiesel and methanol) to a flame, the biodiesel-soaked cotton ball required more time to extinguish and produced more dark ashes.*

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<sup>4</sup> Note that active voice may be used in conjunction with (or in place of) passive voice! (ex - “A reflux was run through the reaction vessel after it was observed that the solution was boiling” is a much more complicated and less direct way of saying the same thing!)

<sup>5</sup> Unlike most lab manuals, the report write-up outlines the methods (or “procedure”) section in paragraph form but retains the same sequence of steps. Depending on instructor expectations, the methods section may also require a materials list and a description of chemical waste disposal. Any variations in steps from the lab manual should be explicitly noted!

<sup>6</sup> In the **Observations**, you build off of the methods by listing any qualitative results or observed *consequences* from the procedural steps. Observations may or may not be necessary depending on how well the procedure was followed and what sort of tests or experimental models were implemented, but they should be *concise* and *precise*!

## Results<sup>7</sup>

$$M_{\text{flask\_empty}} = \underline{84.770 \text{ g}} \quad M_{\text{flask\_after\_seperating}} = \underline{108.731 \text{ g}}$$

## Calculations<sup>8</sup>

$$M_{\text{flask\_empty}} - M_{\text{flask\_after\_seperating}} = M_{\text{biodiesel}} (\text{actual}) = \underline{23.961 \text{ g}}$$

Limiting Reagent: Vegetable Oil (fatty acid trimer)

$$M_{\text{biodiesel (theoretical)}} = \frac{50 \text{ mL V-O}}{1} \times \frac{0.915 \text{ g V-O}}{1 \text{ mL V-O}} \times \frac{1 \text{ mol V-O}}{872 \text{ g V-O}} \times \frac{3 \text{ mol biodiesel}}{1 \text{ mol V-O}} \times \frac{296 \text{ g biodiesel}}{1 \text{ mol biodiesel}} = \underline{46.59 \text{ g}}$$

$$\text{Percent Yield}_{\text{biodiesel}} = \left( \frac{23.961 \text{ g}}{46.59 \text{ g}} \right) \times (100) = \underline{51.43\%}$$

## Conclusion<sup>9</sup> → (OR CAN BE LABELED EQUIVALENTLY AS) “DISCUSSION”

The experiment yielded 51.43% of the available biodiesel product. Viscosity tests of the product revealed that the biodiesel was less viscous than the olive oil. A comparative burn test of the biodiesel product with methanol revealed that the biodiesel burned longer and more time elapsed before the flame extinguished.

Furthermore, the resulting cotton ball containing the burned biodiesel was significantly darker in color than that of the burned methanol. The final yield and qualitative results observed in the two tests are indicative of characteristic

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<sup>7</sup> In the **Results**, data should be presented in a organized linear format, either listed in direct lines or displayed in a figure such as a table or graph depending on the amount of information recorded.

<sup>8</sup> In the **Calculations**, data is mathematically interpreted to yield necessary results for the conclusion. In this case, all calculations were provided but in most experiments - especially those with multiple trials - a sample calculation for each different quantity will suffice.

<sup>9</sup> In the **Conclusion**, you may re-state the important data obtained from the previous section(s), but you should generally omit any calculations, figures, or new empirical information from this section. The purpose of the conclusion is to analyze the existing data, not present new data.

properties and features of biodiesel but as a product that was not fully formed.<sup>10</sup>

The experimental methods employed may have been subject to possible sources of error and should be noted as possible significant factors in the partial synthesis of the product.<sup>11</sup> The physical nature of the very reagents and final product made their complete removal from the reaction vessels difficult. Thus, because of the high viscosity of the substances, all of the glassware utilized during reactions was coated with large amounts of the reagents and also likely the product. Secondly, it should be noted that the boiling temperatures required in the reflux of the final product were not accurately estimated in the methods, causing much of the product to start to boil out the reflux tube. The reaction required a more delicate monitoring of the reaction temperature to ensure no product was lost.<sup>12</sup>

Consequently, despite these considerations of error, the experiment objective was in fact met. The quantitative and qualitative results of Experiment 8 provided direct empirical evidence for the formation of biodiesel via the reflux reaction of sodium methoxide with vegetable oil.

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<sup>10</sup> Be sure to make connections to conceptual material, explaining the specific experimental circumstances in conjunction with a more general principle, reaction, or theory. This section provides the opportunity to demonstrate to your instructor your knowledge of the material and your ability to apply it in the laboratory setting.

<sup>11</sup> Equally as important as explaining your results is giving basis for their outcome. Provide possible sources of error and objectively critique the procedural flaws, noting where the experiment may be improved.

<sup>12</sup> Finishing this section should be a brief evaluation of the experiment in regards to the success of the procedure and expected outcome (i.e. whether or not it met the objective)

#### ADDITIONAL DOCUMENTS THAT WERE HANDED IN WITH THIS LAB REPORT

- Your instructor may require you to submit a number of additional sections and/or documents with this kind of write-up, including:
  - Pre-lab: a brief (one or two paragraph) description of the objective, procedure, and concept material.
  - Post-lab questions: detailed answers to the lab-based and material-based questions given by your instructor at the conclusion of the laboratory period.
  - Lab notes: the data recordings, observations, and hand-written reporting done throughout the experiment itself.
  - Reaction Scheme: if not included in your introduction/objective, these provide a detailed mechanism of the overall reaction occurring amongst the compounds being used. Depending on your instructor, these may or may not be completed already.