

Introduction

Heating chemical reactions by microwave energy has been an increasingly popular tool in the scientific community. Since the first published reports on the use of microwave irradiation to carry out organic chemical transformations by the groups of Gedye and Giguere/Majetich in 1986, more than 3500 articles have been published in this fast moving and exciting field, today generally referred to as microwave-assisted chemical synthesis (MAOS).

Microwave's main advantages are to reduce reaction times, increase product yields and enhance product purities by reducing unwanted side reactions compared to conventional heating methods.

The advantages of this enabling technology have, more recently, also been exploited in the context of multistep total synthesis and medicinal chemistry, drug discovery, and have additionally penetrated related fields such as polymer synthesis, material sciences, nanotechnology and biochemical processes.

In this project; Aspirin and Diels Alder (Preparation of cis-5-Norbornene-2,3-endo-dicarboxylic Anhydride) reactions were placed on PDMS macro arrays to be heated in microwave irradiation. The reactions were synthesized later on by Infra-red spectroscopy. What it has to be concluded at the end of this project is, how long the Aspirin and the Diels Alder reactions take to react using the microwave heat, and what are the products' yields of each reaction.

Methods

5 mmol of Salicylic acid (0.6906 g) and 15 mmol of acetic anhydride (1.5315 g) were mixed and added to the PDMS macro arrays wells, each well composed of 0.1 ml volume. afterwards, the reaction was settled for almost 10-15 minutes, and then it was replaced in the microwave at a remarked place inside the microwave. The aspirin was heated in the microwave at different times; the times chosen were between 1-6 minutes. After the aspirin was heated for 1 minute, then it was taken to be synthesized by infra-red spectroscopy. The same was done after the aspirin was heated for 2, 3, 4, 5, and 6 minutes.

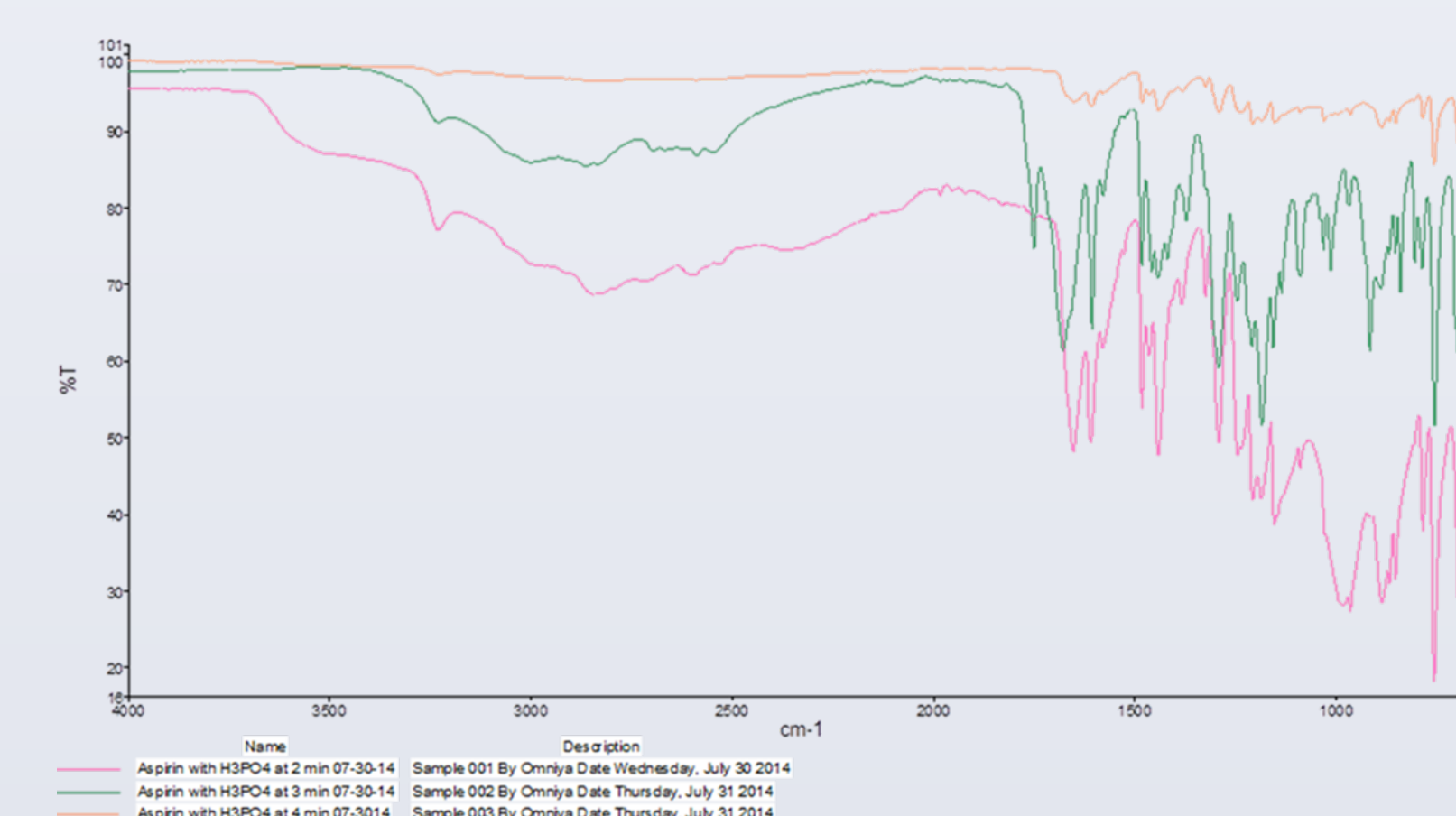
Then, the IR spectra were compared by the commercial aspirin's IR spectra using software called (spectrum), to check if the experimental aspirin's peaks were similar to the commercial one and correct. After this experiment was done, the aspirin reaction was added with phosphoric acid H₃PO₄ (catalyst) in PDMS macro arrays and placed inside the microwave at different times and compared to the original Aspirin's IR spectrum.

For Diels-Adler reaction (cis-5-Norbornene-2, 3-endo-dicarboxylic Anhydride), this product was formed by reacting maleic anhydride and cyclopentadiene. Dicyclopentadiene was distilled by distillation apparatus in the laboratory for approximately 2 hours and 10-15 ml was obtained. Afterwards, the reaction was stirred for 15-20 minutes until colorless solid was participated and the Diels-Alder reaction was pipetted in PDMS macro arrays, which contains 16 wells and each well composed of 0.1 ml. The Norbornene reaction was placed in the microwave at a marked place inside the microwave at different times, between 1-3 minutes. The reaction was then monitored by infra-red spectroscopy and compared to an original spectrum of Norbornene to check if the reaction was obtained.

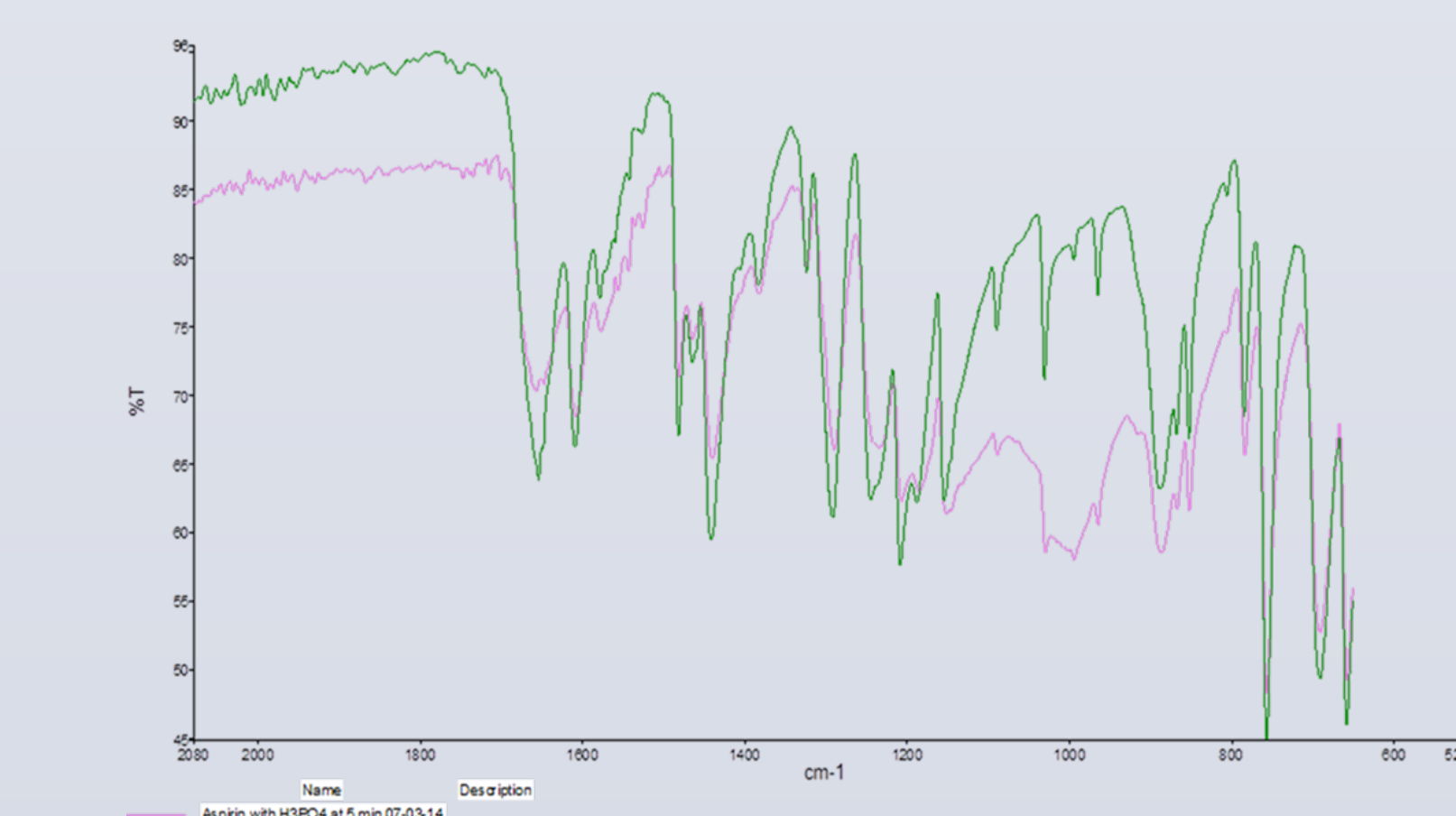
Data

Product	Mass	temperature	Melting point	Percentage yield
Aspirin	2.22 g	50 OC at 5 min	160 OC	95%
Norbornene	0.27 g	60 OC at 5 min	165 OC	97%

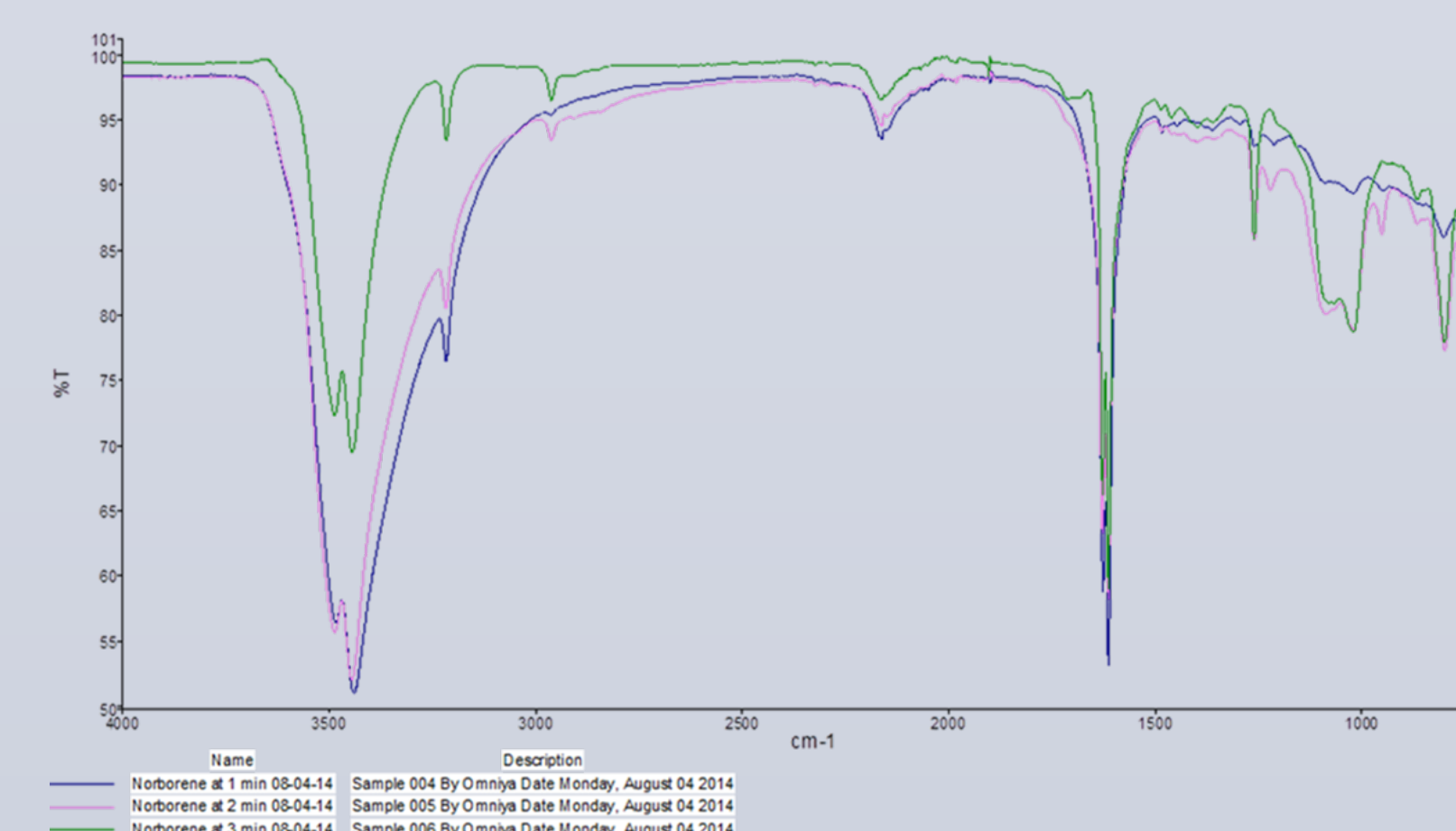
IR spectrum of microwaved aspirin with phosphoric acid H₃PO₄:



Comparison of microwaved aspirin and microwaved aspirin with H₃PO₄ acid:

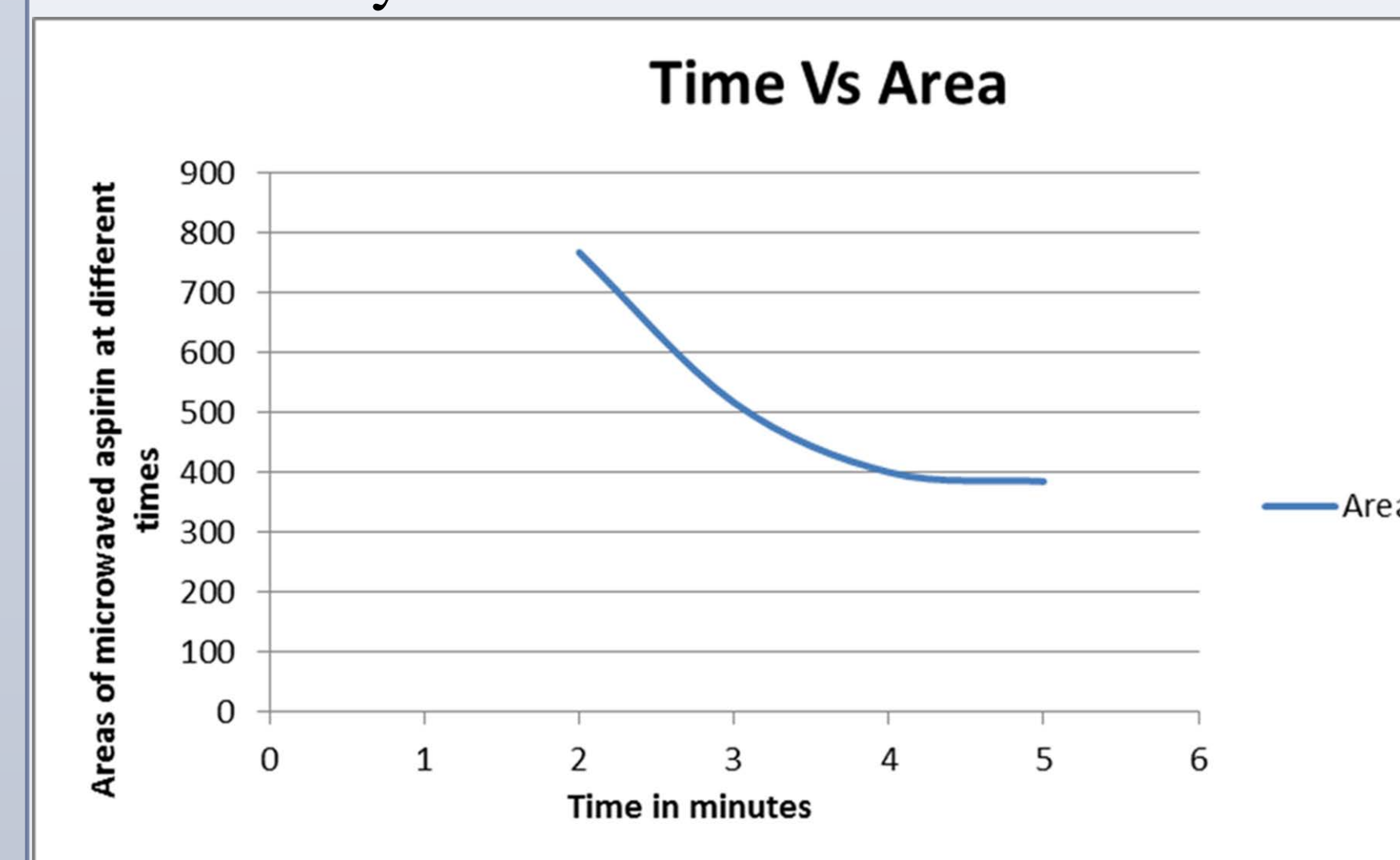


IR spectrum of microwaved Norbornene at different times, between 1-3 minutes:



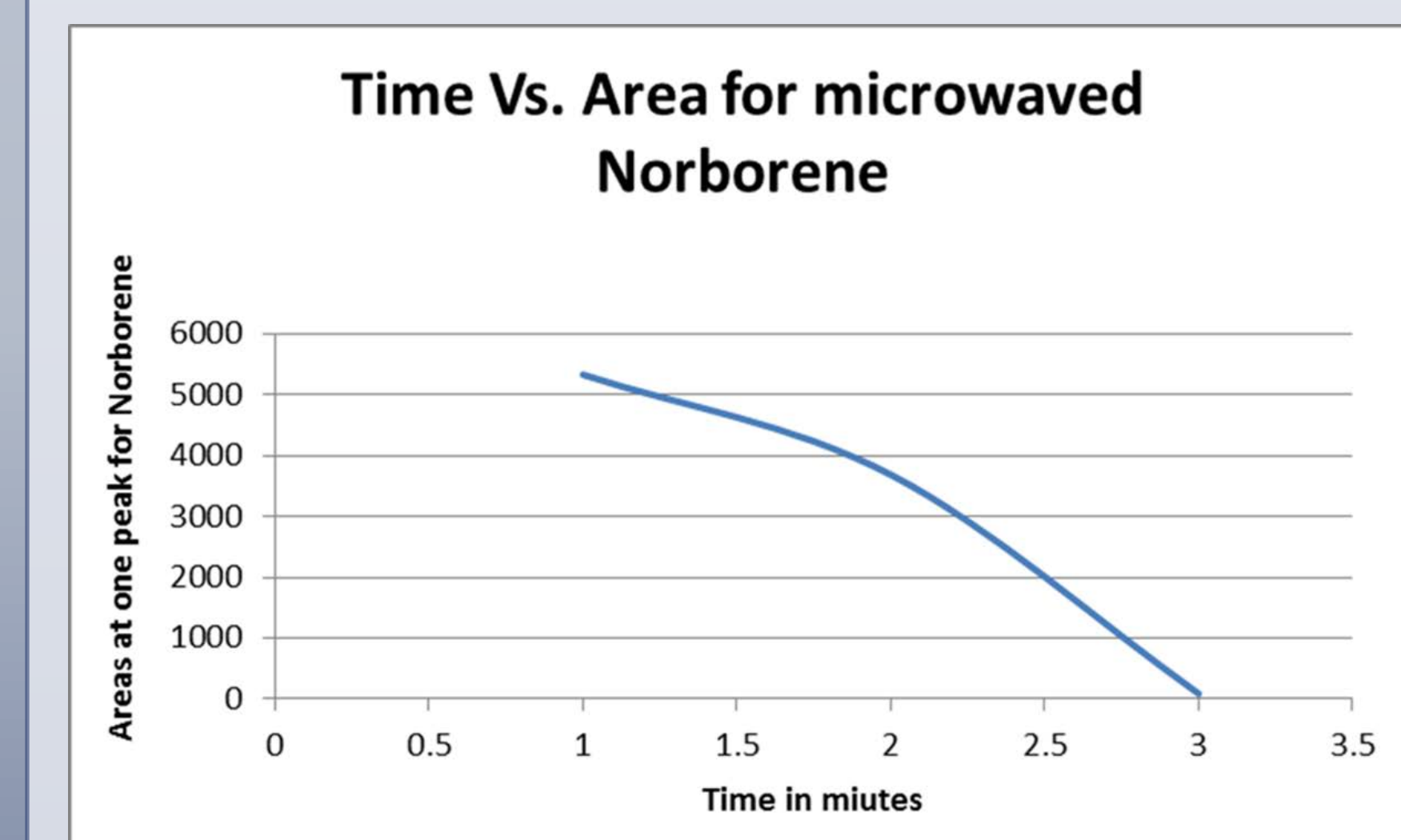
Discussions and Conclusion

A peak was chosen after the syntheses of aspirin to conclude if the reaction was consumed. As the time was increasing the area of this specific peak was decreasing or consuming. As a conclusion, the aspirin was obtained at 5 minutes and the yield increased by 2%.



Discussions and Conclusion

A peak for cis-Norbornene synthesis was chosen to conclude if the area of this specific peak was consuming, as the time increased the area decreased thus, the norbornene was obtained at 3 minutes and the yield increased by 5%.



Acknowledgments

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