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Green synthesis of semicarbazones: A comparison of two green solvents

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Abstract
Semicarbazones are small organic molecules that are frequently used in pharmaceuticals due to their anticonvulsant and anti-tumor biological activity. Previous synthesis of these molecules involved lengthy, high temperature reactions that used toxic and harmful reagents. The focus of this research was to demonstrate the use of a green chemical synthesis in creating these molecules, ultimately optimizing a reaction process for mass-scale production. Green chemistry involves chemical research that is carried out with safe, environmentally friendly reagents in low-energy conditions. Using novel green solvents, an array of semicarbazones were synthesized in an efficient and eco-friendly matter, satisfying green chemistry requirements. The reaction was optimized using two solvents – ethyl lactate and dimethyl isosorbide – both of which qualify as green and are found in cosmetic products. In these two sustainable solvents, we have demonstrated that the reaction can produce quantitative yields of product at room temperature in minutes. These reactions can now be investigated for industrial scale-up, while other small-scale syntheses in these solvents are being developed. The results of this study have promising implications for the development of other green routes to molecules of industrial importance under efficient, environmentally friendly conditions.

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Green synthesis of semicarbazones: A comparison of two green solvents

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Abstract
An array of semicarbazones were synthesized by the reaction of semicarbazide hydrochloride with substituted benzaldehydes. The reactions were performed in two different novel green solvents, ethyl lactate and dimethyl isosorbide. The yields and purities of each product were determined to be greater than or equal to 80% for each reaction performed using H NMR spectroscopy as well as LC-MS. The results indicate that the reaction is most efficient when the ratio of ethyl lactate:water is 80:20, and the ratio of dimethyl isosorbide to water is 92:8. Generally the semicarbazones synthesized in ethyl lactate displayed a higher yield and degree of purity.

Methods & Results

Synthesis of Semicarbazones: Solutions containing 100%, 90%, 80%, 70%, and 60% ethyl lactate in water were prepared as solvents. This process was repeated for dimethyl isosorbide solutions of concentration 100%, 98%, 96%, 94%, 92%, and 90%. A semicarbazide standard was also prepared by creating a 1mmol solution in water. For the reaction, 1mmol of a substituted benzaldehyde was dissolved in 0.3mL of the solvent, which varied in concentration for each trial. 0.25 mL of the semicarbazide solution was then added to this mixture, and the reaction proceeded in a reaction tube. The mixture was vortexed, and the time to precipitation was recorded. Eventually the reaction tube was placed in a freezer to aid crystallization. The products were filtered using a Hirsch funnel, washed with distilled water, and dried in a vacuum oven for 48 hours. Finally, the dried products were weighed and yields were calculated. The semicarbazide-benzaldehyde solutions in 80% ethyl lactate and 92% dimethyl isosorbide solvents produced the best yields.

LC-MS: Semicarbazones produced in 80% ethyl lactate and 92% dimethyl isosorbide were prepped for LC-MS analysis using 60% acetonitrile as a solvent, and analyzed using a Agilent 1260 infinity™ spectrometer. Liquid chromatography tested for purity and mass spectrometry confirmed the completion of the reaction. Vanillin semicarbazone is shown in Figures 3 and 7.

Discussion & Conclusions
The results of the syntheses indicate that the best yields were in the presence of an 80:20 ethyl lactate:water mixture and a 92:8 dimethyl isosorbide:water mixture. The results of the NMR analysis show that the products made fit the expected structure. In addition, the LC-MS analyses indicated a high degree of purity, where only one product peak appeared with only small traces of starting material detected. The mass spectrum also proved the structure of the compounds by providing the expected molecular ion peak and fragmentation pattern. It can therefore be concluded that the proposed synthesis works in two novel green solvents, the products of which are high yielding and highly pure. Future study will investigate the scaling up of this reaction for industrial use, as well as the use of other green solvents.

References