https://doi.org/10.33805/2641-7383.126
Volume 4 Issue 1 | PDF 126 | Pages 3
Edelweiss Chemical Science Journal

Editorial ISSN: 2641-7383

Attempt to Reduce Potentially Flammable Organic Solvents in Chemical Synthesis

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Citation: Akitsu T, Onami Y and Furuya T. Attempt to reduce potentially flammable organic solvents in chemical synthesis (2021) Edelweiss Chem Sci J 4: 16-18.
Received: Jun 08, 2021
Accepted: Jun 25, 2021
Published: Jun 29, 2021

Abstract

A fire sometimes ignited an organic solvent in a chemical laboratory. A mechanochemical synthesis may be a good way to reduce the amount of organic solvents or without organic solvents compared to conventional synthesis in solutions. The solvent affected the product, namely solvent methanol acted as a ligand, although the quality of the data of X-ray crystallography is usually difficult to report in a strict research article in the case of our Azo-Schiff base metal complexes. Thus substitution of synthesis methods associated with potentially dangerous organic solvents may be possible depending on the application or purpose.

Keywords: Organic solvent, Ignition point, Schiff base metal complexes, Mechanochemical synthesis, Crystal structure analysis

Abbreviations: THF-Tetrahydrofuran, KOH-Potassium Hydroxide, $Cu(OAc)_2$-Cupric Acetate

Introduction

Some chemical substances are dangerous from the viewpoint of fire or explosion [1-3]. A database containing the causes of fire accidents will be useful for the safety and education of chemical processes [4,5]. For example, occupational accident data were collected for many years and constructed an explosion and fire database, which described the contents of the disclosure and the outline of the extraction/search method of cases, and also introduced the transition of chemical process disasters and analysis examples by industry [4].

As stated in accident reports in 2010's (more than ten years ago), fire in a chemical laboratory sometimes occurred by igniting organic solvents (flash and ignition points/°C) such as methanol (11, 646), n-hexane (-22, 225), and Tetrahydrofuran (THF) (-17, 321) [6,7].

- When carrying out recrystallization using an *organic solvent (unreported)*, a beaker containing the solution was heated on an open flame and ignited, causing a small fire.
- When an Erlenmeyer flask with methanol in it was placed in a heating-type constant temperature water bath, water in the water bath was depleted, the empty water bath was heated and its temperature became high, and then the *methanol* ignited.
- While heating *methanol* in a fume hood, an experimenter returned to his or her room. A break occurred, and the *methanol* ignited. An organic solvent close by also ignited, and the fume hood burned.
- 3,5-di-t-butylbenzonitrile was put in a conical beaker in a fume hood to recrystallize it, *n-hexane* was added to the beaker, it was covered with aluminum foil, and heated on a hot plate while stirring it with a magnetic stirrer. Bumping of the solvent occurred, and it ignited. The experimenter burned his or her face and right arm during the ignition.
- During distillation of *THF anhydrous*, the inside pressure was increased, and the *THF* leaked and was ignited by a mantle heater. The ground glass joint came off, and the *THF* inside was also ignited, causing a large fire in which the flames reached the ceiling.
- An unattended reaction using *THF* at night caused an explosion and flames. A person outside of the university noticed that the laboratory window was bright red and reported it to the fire department.
- While distilling *THF*, a sudden explosion occurred, and a fragment of the flask penetrated the experimenter's heart, which resulted in instant death.
- A flask suddenly exploded during the reaction using *THF* as a solvent, and the burned *THF* was exposed to the experimenter's face. The face was restored by plastic surgery, leaving a keloid on the entire face of the experimenter.
- When a glass stopper was taken to add benzophenone during purification and distillation of *THF*, *THF* boiled rapidly and spouted, ignited, and exploded in the fume hood exhaust pipe. The experimenter burned his or her face and upper arms.
- When *THF* was constantly distilled, a flask was cracked, and THF leaked. The container was almost empty.
- When attempting to distill *THF*, an experimenter mistakenly turned on the power of the heating device of the ether distillation unit (no reflux pipe connected) next to it, and it exploded.

In order to avoid accidents due to organic solvent like them, a mechanochemical synthesis may be a good way to reduce the amount of or use of organic solvent compared to conventional synthesis in solution. So we attempted to compare conventional solution method and wet mehanochemical method to reduce methanol solvent for azo-containing amino acid derivative Schiff base copper(II) complexes [8,9].

Experimental

- **Conventional solution method**: In an Erlenmeyer flask (200 mL), methanol solution (100 mL) of *L*-phenylalanine (0.033 g, 0.20 mmol), KOH (0.011 g, 0.20 mmol), and azo-salycylaldehyde (0.045 g, 0.20 mmol) was stirred for 3hr at 40 ^{O}C. $Cu(OAc)_2 \cdot H_2O$ was added and stirred for 3h and filtrated and dried **(Figure 1).**
- **Wet mehanochemical method:** In a mortar containing a small amount of methanol, *L*-phenylalanine (0.033 g, 0.20 mmol), KOH (0.011 g, 0.20 mmol), and azo-salycylaldehyde (0.045 g, 0.20 mmol) were added and grind for 5 min at room temperature to give red product. $Cu(OAc)_2 \cdot H_2O$ was added and grind for 5 min to give yellow-green product and washed with water and dried **(Figure 2).**

Results and Discussion

The products were confirmed X-ray single crystal structure analysis **(Figure 3).** The solvent affected the products. Water molecules in methanol solvent or hydrate ligand coordinated to copper for the conventional method, while similar water and methanol molecule of small amount of solvent for grinding coordinated to copper for the wet mechanochemical method. However, the quality of the resulting data of X-ray crystallography was quite poor for both cases **(Figure 4).** Unfortunately, in a normal standard, we cannot repot this result as an original research article in any journals.

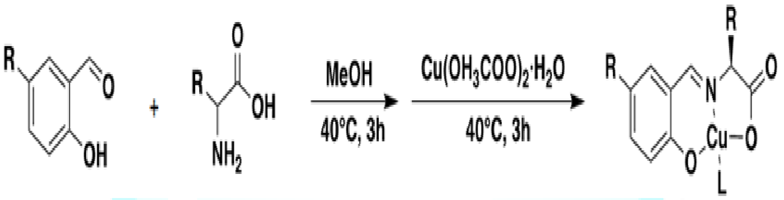

Figure 1: General synthesis reaction of *L*-amino acid derivative Schiff base copper(II) complexes in a methanol solution [8].

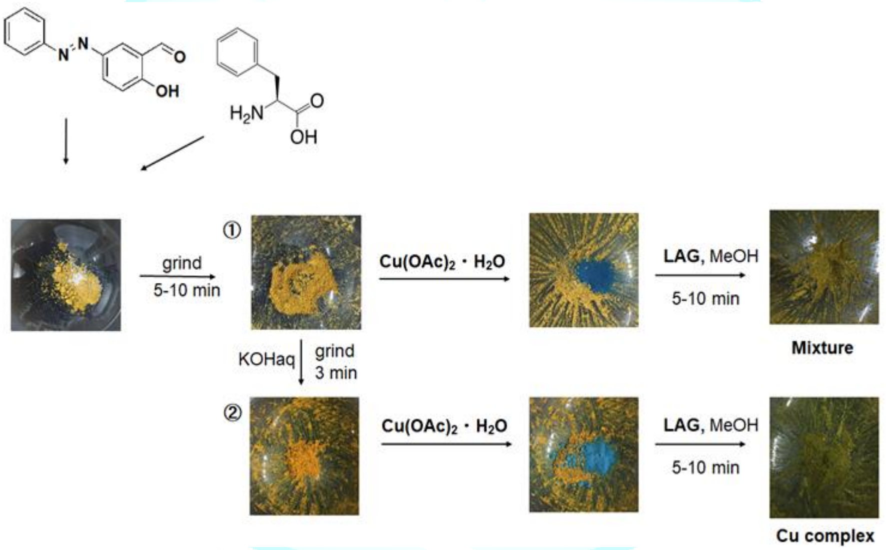

Figure 2: Reaction steps of the azo-containing *L*-amino acid derivative Schiff base copper(II) complexes with photos of each step (Liquid-Assisted-Grinding is abbreviated as LAG). The steps ① and ② may correspond to condensation and deprotonation, respectively.

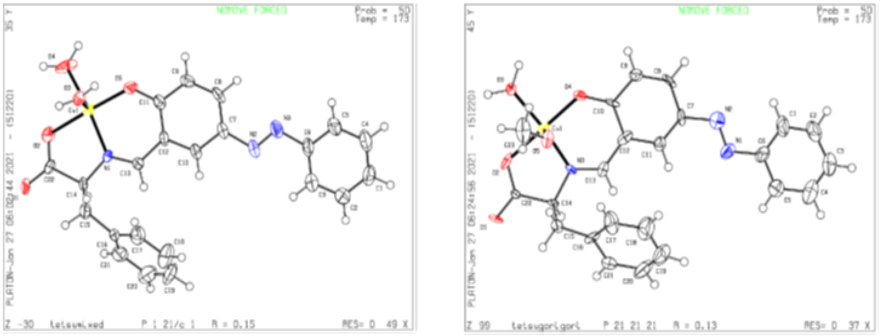
(Solution) (Wet mechanochemical)

Figure 3: Tentative crystal structures (PLATON drawings) of the products from (left) solution and (right) wet mehanochemical method.

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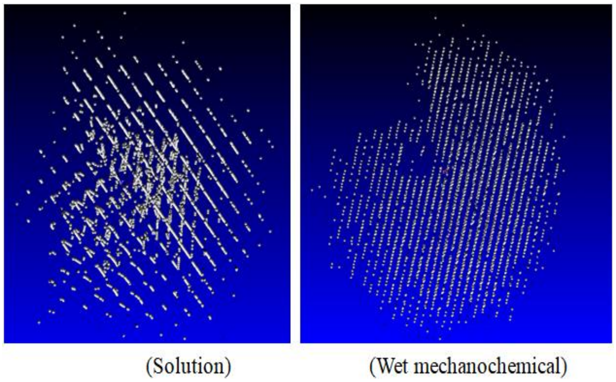
(Solution) (Wet mechanochemical)

Figure 4: Raw data of X-ray diffraction of single crystal of the two products; (left) twin or additional microcrystals for solution, (right) low resolution for wet mehanochemical method.

Conclusion

In this way, substitution of organic solvents (to use higher flash and ignition points but required similar polarity or solubility) as well as synthesis methods associated with potentially dangerous organic solvents may be possible depending on the application or purpose. Even if it is a finding that cannot be made into a formal academic paper, we would like to inform you as a safety measure and know-how within the research group, so we wrote it here to contribute to the safety of chemists and the development of chemistry.

Acknowledgement

This work was supported by a Grant-in-Aid for Scientific Research (A) KAKENHI (20H00336).

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