Reaction Engineering Approach for Designing an Impinging-Jet Reactor for Meerwein Arylation

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Highlights

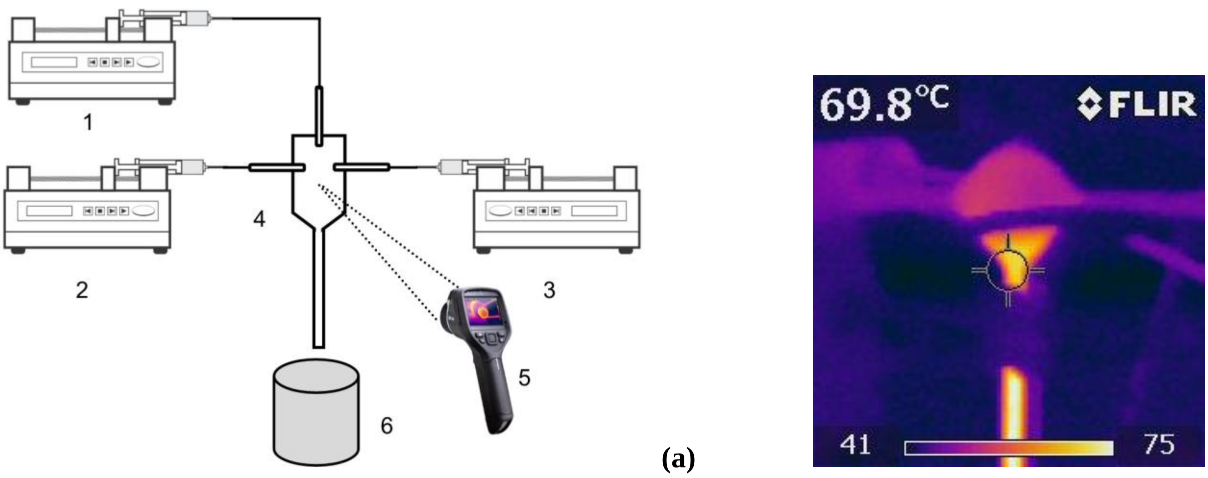
- In-situ diazotization and meerwein arylation.
- Hot spot temperature monitoring using IR camera.
- Using impinging jet reactor for high throughput.
- The throughput of the impinging-jet reactor was 43 times higher than the batch reactor.

1. Introduction

Meerwein arylation is a useful transformation in pharmaceutical[1] and Agro industries[2]. Traditional batch mode operation suffers from many safety issues like vigorous N_2 evolution causing froth or foam, handling unstable diazonium salts and heat management. Recently, flow chemistry has enabled the researchers to perform meerwein arylation in a continuous mode[3]. Here we report a novel method for continuous meerwein arylation which can also be used for higher throughput.

2. Methods

Figure 1(a) shows the experimental setup for in-situ diazotization and meerwein arylation in an impinging-jet reactor. The reactor has three inlets and one outlet. The top inlet is used for passing sodium nitrite solution and the side inlets are used to pass the solution containing a mixture of 3,4-dichloroaniline, hydrochloric acid, acetic acid, methacrylic acid and cuprous chloride. The reaction mixture was quenched at the outlet using ice and/or β-naphthol and sodium hydroxide solution in a well-mixed beaker. IR camera was used to capture the hot spot temperature (see Figure 1. (b)).



(a) **(b)**

Figure 1. (a) Experimental Setup for Impinging Jet reactor. (1) Syringe pump containing sodium nitrite solution, (2) & (3) Syringe pump containing a mixture of 3,4-dichloroaniline, HCl, methacrylic acid, acetic acid, CuCl, and acetone, (4) Impinging jet reactor, (5) IR camera, (6) Beaker for quenching. (b) Thermal image of impinging jet reactor captured using FLIR i5 camera

3. Results and discussion

Initial batch experiments were performed to optimize the reaction yield. It was observed that increasing catalyst quantities would accelerate the reaction rate by many folds. The mole ratio of the methacrylic acid (1.5 eq. to 3 eq.) and the catalyst quantity (0.5 mol% to 5 mol%) was varied to optimize the yield of the batch reaction. The initial temperature before catalyst addition was 20°C for each case and the maximum temperature during the reaction varied from 22 ^{0}C - 51^{0}C depending on the catalyst quantity. The isolated yield was in the range of 40% - 77.21%. It was observed that the yield of meerwein arylation was highly dependent on temperature. After optimizing the reaction in batch mode, experiments were performed in an impinging jet reactor (see Figure 1(a)) at a flow rate ranging from 34 ml/min to 64 ml/min (Re~722-1360). The feed solution was at ambient temperature and the measured hot spot temperature was in the range of 41.5^{o}C - 70.1^{o}C (see Figure 1 (b)). The yield of 75.7% was obtained in the impinging-jet reactor with a throughput of 88 g/hr. Currently, kinetics experiment and optimization studies are under progress. CFD simulations are also in progress to improve the mixing by optimizing the reactor geometry and study the temperature profile.

4. Conclusions

Meerwein Arylation was performed in a batch reactor and impinging-jet reactor to obtain 77.21% and 75.7% yield respectively. However, throughput in the impinging-jet reactor was 43 times higher than the batch reactor. Further experiments in the impinging jet reactor were performed and the hot spot temperature was monitored using IR camera. The results suggest that controlling the hot spot temperature by varying the feed inlet temperature will enhance the selectivity. Currently, kinetics and optimization studies are under progress. Furthermore, CFD simulations are in progress for studying the mixing process which will be coupled with heat transfer and reaction to study the temperature effects on the selectivity.

References

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Keywords
Meerwein Arylation; Impinging-jet reactor; IR monitoring; Hot spots