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1   **Investigating the effect of increasing cloth size and cloth number in a Spinning Mesh Disc Reactor**

2   **(SMDR): A study on the reactor performance**

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8

9   **Abstract**

10   The spinning mesh disc reactor (SMDR) is a rotating catalytic reactor with a potential to facilitate process

11   intensification. In this study, the scale-up of a newly designed SMDR has been demonstrated by increasing (i)

12   cloth size and (ii) cloth number for tributyrin hydrolysis and nitroaldol condensation reaction. The effect of

13   spinning speed, cloth size and cloth number was investigated using design of experiments and the results show

14   an increase in the cloth size or cloth number leads to a higher reaction rate. This is due to (i) an increased

15   catalyst loading with increase in surface area and volume of the cloth stack and (ii) reduced film thickness

16   with increasing shear forces and longer residence times improving the overall mass transfer. Addition of

17   multiple cloths of increasing cloth sizes further improved the reaction rates at higher substrate concentration.

18   A maximum reaction rate of 6.9 mM min^{-1} and 0.043 mmol min^{-1} was obtained for three 50 cm cloths for

19   tributyrin hydrolysis and nitroaldol condensation reaction respectively. These results highlight the potential

20   routes for the SMDR scale-up without a loss in the reaction efficiency for a range of catalytic reactions, thus

21   allowing for a tuneable operation of the SMDR for industrial applications.

22   **Keywords**

23   Spinning mesh disc reactor; reaction scale-up; increased catalyst loading; multi-cloth stack; reaction

24   intensification

25

26

1. Introduction

Scale-up of traditional reactors is straightforward but accompanied by high mass and heat transfer resistance impeding the reaction rate and reduced product yield. Hence, careful consideration of the reaction system is necessary to ensure a sustainable process scale-up as they often involve large quantities of solvents and expensive catalysts. This can be overcome by achieving a better control over the reaction at a molecular level and follows a new development path in chemical processing known as process intensification (PI) [1]. PI can be achieved either by modifying the reactor design or employing innovative reaction techniques. One of the more popular approaches of PI is to achieve a reduction in the size of the chemical equipment or simplify its scale-up whilst maintaining the production efficiency [2]. This has led to the concept of 'numbering-up', where process scale-up is achieved by adding more reactor units in series or parallel. Generally, numbering-up reduces the reactor size, promoting safe operating conditions by confining the operating area to a smaller space, especially for reactions involving hazardous chemicals.

The Spinning disc reactor (SDR) is an intensified reactor which uses centrifugal force to drive the reaction fluid to a thin film of high shear on the surface of the spinning disc[3]. Scale-up in the SDR has been demonstrated by either increasing the disc size or by adding more discs ('numbering-up') to the central shaft. Scale-up by increasing the disc size in the SDR has been achieved for production of polyurethane [4]. A modification of the SDR is the rotor stator spinning disc reactor, where it has been shown that addition of multiple rotors to the central axis resulted in improved mass and heat transfer coefficients for multi-phase and catalytic reactions [5, 6]. The spinning mesh disc reactor (SMDR) is another variation of the SDR, which additionally houses a mesh cloth support with immobilised catalyst on the disc. The reaction liquid impinged on to the centre of the spinning disc allows for the formation of a thin film of high shear over and within the cloth surface, facilitating rapid liquid mixing and enhanced mass transfer. Further, residence time distribution studies has shown that addition of cloths in the SMDR causes a significant deviation from the plug flow behaviour reported for a conventional SDR, indicating that the flow property resembles a well-mixed reactor [7].

Reaction intensification has been demonstrated by using catalysts immobilised on the wool for: (i) lipase catalysed hydrolysis of tributyrin [8-10], (ii) Henry reaction catalysed by copper triflate [11] and (iii) kinetic

resolution of racemic alcohol catalysed by amano lipase [12] and improved reaction efficiency was achieved in all the cases compared to the batch reactor. Process scale-up in the SMDR has been achieved through the concept of numbering-up by addition of more catalyst cloths on to the disc. Significant improvement in the reaction rate and productivity (g l^{-1} h^{-1} of product formed) has been observed for the systems described above with the increase in the number of catalyst cloths. This demonstrates that the transition of SMDR from a laboratory process to an industrial scale operation is accompanied by minimum alterations to the original reactor design without compromising on the overall process efficiency. However, the studies till date have only been carried out with a maximum cloth size of 25 cm and a comprehensive scale-up study for increasing cloth diameter and numbering-up of larger cloths for a range of reactor parameters has not been explored.

The aim of this study is thus to investigate and compare the performance of the SMDR when scale-up is achieved by (i) increasing the catalyst cloth size and (ii) numbering-up of cloths (of similar and increasing diameters). Two different reactions: enzymatic hydrolysis of tributyrin (aqueous system) and nitroaldol condensation reaction catalysed by copper triflate (organic solvent system) have been chosen for this study as we have previously optimised both reactions in the SMDR and they have also shown potential for application in pharmaceutical and fine chemical industry. The reactions will be carried out in a newly designed reactor with an optimised nozzle design to minimise the spin-up zone and also improve the contact between the reactant and the cloth surface. The effect (individual and simultaneous) of increasing cloth diameter (20 cm to 50 cm) and number of cloths (one to three) on the reaction rate will be investigated using design of experiments. The reactor performance will also be examined as a function of film thickness and mean residence time.

2. Material and methods

2.1 Materials

All of the chemicals and lipase from *Pseudomonas fluorescens* was obtained from Sigma Aldrich and used as received. Saskatoon knitted woollen blanket (cream colour, 1.5 mm thick, 0.32 g cm^{-2}) was obtained from Urbanara (Berlin, Germany). Deionised water was used to prepare all standard solutions and buffers.

*2.2 Immobilisation of lipase on wool*

80 The general protocol used for lipase immobilisation on wool is as reported in [10], with modifications to

81 reagent concentrations based on the weight of the woollen cloths used in the present study. To summarise,

82 circular pieces of different diameters (20 cm, 30 cm and 50 cm) were cut from a woollen blanket cloth. Pre-

83 treatment of the cloths was carried out using a solution of sodium silicate and hydrogen peroxide in a pH 9

84 buffer for 70 minutes. The cloths were then soaked in a solution of 2% polyethyleneimine (PEI) at pH 8 for 2

85 hours and washed with deionised water, after which they were soaked in a solution containing 2 gL^{-1} lipase in

86 a pH 6 phosphate buffer for 24 hours. Finally, the cloth was dipped in a 0.5% glutaraldehyde solution in pH 6

87 phosphate buffer for 10 minutes to facilitate enzyme cross-linking and rinsed with deionised water to remove

88 any unbound enzyme from the cloth surface.

89 *2.3 Copper triflate immobilisation on wool*

90 The detailed protocol has been published in our previous publication [11]. The procedure for copper

91 immobilisation is similar to that of lipase on wool. The woollen cloths after the PEI treatment were soaked in

92 copper triflate solution (1mM) in methanol for 24 hours. The crosslinking of copper cloths were carried out

93 in a similar manner to that of lipase cloths.

94 *2.4 Reactions in the SMDR*

95 As shown in Fig.1, the SMDR consists of a circular disc fastened with the catalyst cloth and connected to a

96 central rotating shaft within a circular housing. The spinning speed is controlled via a variable speed controller

97 and the disc is driven by a three phase AC motor. The inlet reaction liquid from the reactant vessel is pumped

98 through the central rotating shaft and through a nozzle located at the centre of the disc. The liquid at the centre

99 of the disc has the same speed as the spinning disc, hence minimising the spin-up zone from the centre of the

100 disc when the reactant comes in contact with the disc. This maximises the area of contact between the cloth

101 and the reactant, ensuring a uniform coverage of the cloth. The spin-off from the edge of the disc passes

102 through circular ports located around the casing and into a reservoir from where it is pumped back into the

103 reaction vessel. The disc and the housing are made of hard anodised aluminium and the inlet port tubing is

104 stainless steel, to improve the overall solvent resistance of the reactor.
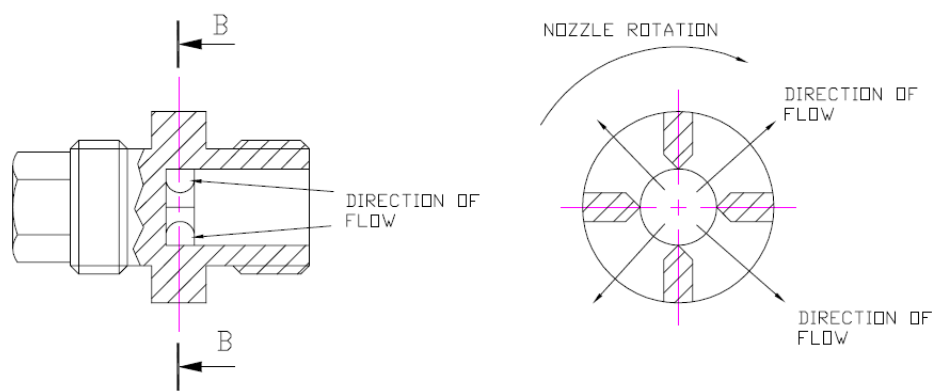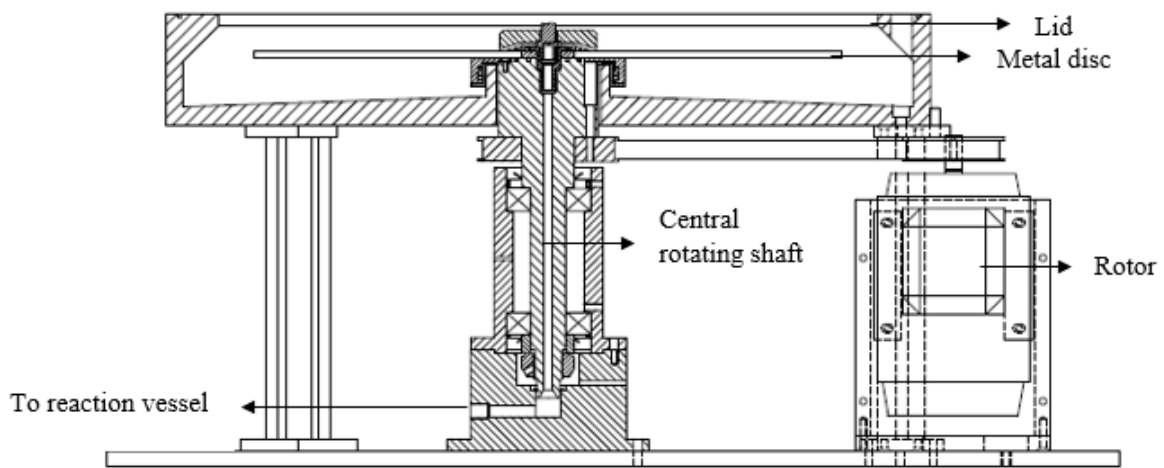
(a)

(b)

Figure.1 (a) Schematic diagram of the SMDR and nozzle design and (b) photograph of the SMDR

(a) Tributyrin hydrolysis in the SMDR

120 An emulsion of tributyrin was prepared by adding Triton X-100 (3.5 gL^{-1}) and tributyrin to 1000 ml of pH 7

121 phosphate buffer (0.1M) such that the final concentration of the reagents were and 33 mM respectively and

122 Fig 2 shows the reaction scheme.

123 Tributyrin   Glycerol   Butyric acid
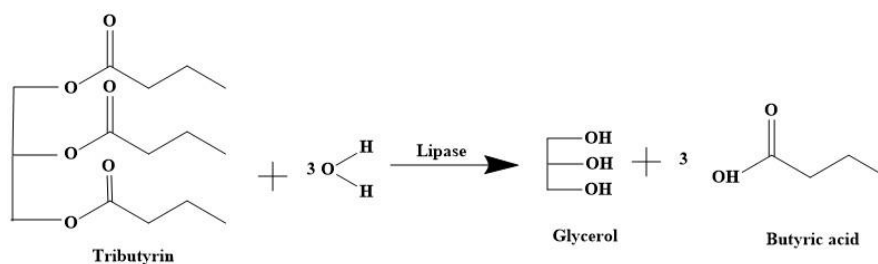
124 Figure.2 Reaction scheme for enzymatic hydrolysis of tributyrin

125 The mixture was emulsified at 1000 rpm for 10 minutes. Lipase cloth of the desired size was fastened to the

126 disc using the screw at the centre of the reactor and the reactor was covered with a Perspex lid. The reaction

127 mixture was fed to the reactor via a peristaltic pump at a flowrate of 3 ml s^{-1} and rotated at the desired speed.

128 The reaction was run for 3 hours at 45°C. A pH stat (Titroline, SI analytics) was used for continuous addition

129 of sodium hydroxide to maintain a constant pH and the data was recorded continuously onto a PC using a

130 hyperterminal software. The reaction conversion was determined as follows [9]:

$$Conversion(\%) = \frac{moles\ of\ free\ butyric\ acids}{moles\ of\ original\ esters\ in\ tributyrin} \times 100$$

131

132 The initial reaction rate was calculated from the slope of the hydrolysis curve obtained from the pH stat. All

133 data points were used from the start of the reaction till the slope began to decrease, which indicates the end of

134 the initial rate period [13].

135 (b) Nitroaldol condensation reaction in the SMDR

136 The reaction mixture consisting of benzylamine (1 mmol), trimethylamine (0.16 mmol), nitromethane (10

137 mmol) and dodecane (0. 5mmol, internal standard) was dissolved in 500 ml ethanol and Fig.3 depicts the
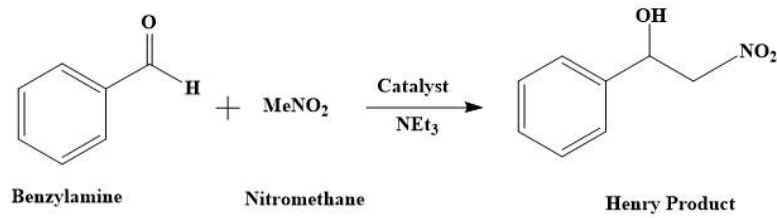
138 reaction scheme.

Benzylamine   Nitromethane   Henry Product

139

140   Figure.3 Reaction scheme for nitroaldol condensation (Henry) reaction

141   The copper catalyst cloth was fastened to the disc in the reactor and the reactor was covered with a Perspex

142   lid. The reaction was initiated by switching on the peristaltic pump once the reactor has reached the desired

143   RPM. The reaction was carried out at 25°C for 3 hours. Samples were taken every 5 minutes during the first

144   30 minutes of the reaction and thereafter for every hour till the end of 3 hours. The samples were analysed

145   using gas chromatography (GC) using a method described in our earlier publication [11]. The first order

146   reaction rate was calculated for the first 60 minutes of the reaction time period.

147   The average surface shear ($\bar{S}$) was used to characterise the reactor performance based on the cloth size and the

148   spinning speed for both reactions as follows [9]:

149
$$\bar{S} = \frac{1}{R} \int_0^R S dr = \frac{3}{4} \left(\frac{3QR\omega^4}{2\pi v^2} \right)^{1/3} \quad (1)$$

150   where, S = surface shear (s^{-1}); Q = volumetric flow rate (m^3 s^{-1}); R = radius of the disc (m); ω = angular

151   velocity (rad s^{-1}); v = kinematic viscosity (m^2 s^{-1}). A maximum spinning speed of 450 RPM was used for all

152   experiments to operate the reactor below the critical shear stress value.

153   The resulting film thickness (f) as a function of the spinning speed was calculated using the following equation

154   [14]:

156
$$f = \left(\frac{3\mu Q}{2\pi\rho R^2 \omega^2} \right)^{1/3} \qquad (2)$$

157

155   where, f = film thickness (m); μ = liquid viscosity (Pa s)

158   The mean residence time (t) of the liquid feed on the cloth surface was calculated as follows [14]:

159
$$t = \left(\frac{81\pi^2 v}{16\rho Q^2 \omega^2} \right)^{1/3} R^{4/3} \qquad (3)$$

160   Where, t = mean residence time (s). The residence time for 'n' number of cloths was calculated by multiplying

161   the residence time for a single cloth with the cloth number (n).

2.5 Design of Experiments (DoE)

163 The effect of different factors such as spinning speed, cloth number and cloth size on reaction conversion and

164 rate was analysed using the Design of Experiments (DoE) approach. Minitab statistical software was used to

165 create a two-level full factorial design and the experiments were performed in random order. The response as

166 a function of conversion and rate were analysed using an Effects and a Pareto chart plot. The standardised

167 effect of the factors were plotted according to their order of significance in the Pareto chart and the dotted line

168 indicating a significance of 5%.

169 **3. Results and discussion**

170 *3.1 Effect of increasing cloth size and cloth number on the enzymatic hydrolysis of tributyrin in the SMDR*

171 **Design of Experiment**

172 The results from DoE are summarised in Table 1 and discussed with respect to the reaction rate as the response

173 as it incorporates more data points during the course of the reaction. The hydrolysis reaction occurs in two

174 steps. The first, faster stage is the hydrolysis of the first ester bond in the water phase and available readily for

175 the lipase. The reaction rate decreases thereafter due to the steric hindrance caused by the remaining ester

176 bonds, and are thus not easily accessible for the lipase. At around 50% conversion, the reaction solution turns

177 from a cloudy emulsion to a clear solution [15, 16]. As most of the reaction occurs in the first 60 minutes, the

178 initial reaction rate has been used to characterise the effect of operating parameters on reactor scale-up.
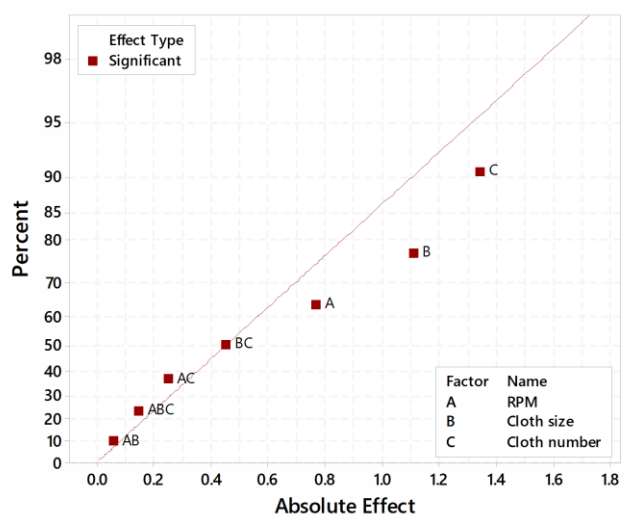
179 As can be seen from Fig. 2 (a) and (b), the normalised effects plot and the Pareto plots for reaction rate as the

180 response indicates that cloth number, cloth size and RPM (in this order) have a significant effect on the

181 conversion. In the effects plot, the factors that are farthest away from the origin have a greater significance on

182 the response. The Pareto chart is a plot of factors in the order of significance of their standardised effects (from

183 the effects plot) on the initial reaction rate with the dotted line indicating a significance of 5%.   Increasing

184 cloth number and cloth size individually has a greater effect on the reaction rate compared to increasing both

185 variables at the same time (explained in the following section). Spinning speed on its own also has a positive

186 effect on the reaction rate, but less significant compared to cloth size and number. This is because the

187 enzymatic reaction is operated at a spinning speed which corresponds to that below the critical shear stress to

188 minimise enzyme deactivation [9, 12]. Overall, the results from the DoE studies indicates that reaction scale-

189 up in the SMDR primarily depends on either the cloth size and cloth number and increasing multiple factors

190 at the same time does not have a significant effect on the reaction rate for a given substrate concentration.
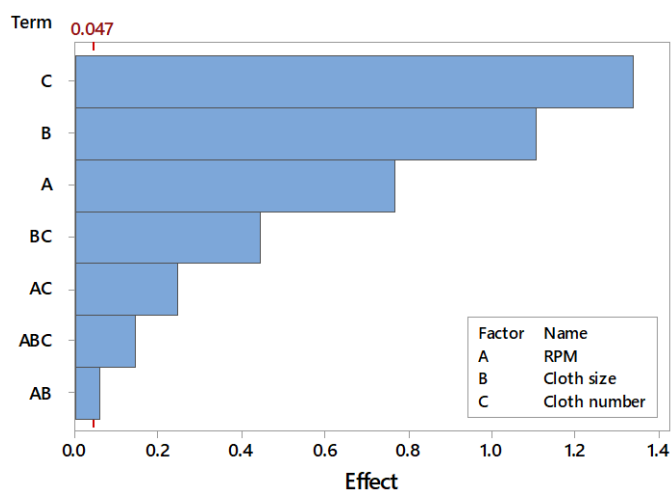
191 Table 1. Results from the Design of Experiments studies for tributyrin hydrolysis

Entry	Spinning Speed (RPM)	Cloth diameter (m)	Cloth number	Initial reaction rate (mM min^{-1})	Conversion (%)
1	450	0.5	1	5.03	87
2	250	0.2	1	2.46	67
3	450	0.2	1	3.56	75
4	250	0.5	1	4.10	83
5	250	0.5	3	5.10	85
6	450	0.2	3	4.96	82
7	450	0.5	3	5.82	91
8	250	0.2	3	4.64	79

192

193

194   (a)   (b)

195   Figure 4: DoE plots with reaction rate as response: (a) Effects plot, (b) Pareto chart plot

196 **Effect of cloth size:** An increase in the cloth size results in an increase in the overall surface area of the cloth

197 and also the catalyst loading for a given substrate concentration. Fig. 3(a) shows an increase in the reaction

198 conversion from 67.6% to 87.7% after 3 hours and fig 3(c) shows an increase in the reaction rate from 2.67mM

199 min^{-1} to 4.64 mM min^{-1} with an increase in the cloth size from 20 cm to 50 cm. Fig 3(b) shows that an increase

200 in the cloth radius results in a lower mean film thickness due to increasing average surface shear (equation 2).

201 This results in rapid mixing within the thin film of liquid over the cloth surface resulting in lower mass transfer

202 resistance and better contact with the substrate and the catalyst. Hence, the mean film thickness is an additional

203 factor that results in higher reaction rates with an increase in the cloth size.

204 **Numbering-up of cloths:** Figure 3 (c) shows the effect of numbering-up catalyst cloth on reaction rate and

205 the reaction rate increased with addition of cloths across the various cloth sizes. The reaction rate for three 20

206 cm cloths (4.64 mM min^{-1}) is comparable to the reaction rate obtained using one 50 cm cloth (4.38 mM min^{-}

207 ^{1}). Addition of cloths results in an increase in the total volume of the cloth stack and hence the enzyme loading

208 (supplementary information A). Visual studies have shown that the centrifugal and gravitation forces promote

209 complete wettability of the cloth stack in both tangential and downward direction facilitating reaction both on

210 and within the catalyst cloth surface [7]. The increase in the reaction rate for multiple 20 cm cloths is more

211 pronounced compared to multiple 30 cm and 50 cm cloths at a lower substrate concentration. This is because,

212 the catalyst: substrate ratio is high even with a single large cloth and the reaction goes to completion at a faster

213 rate and no further increase in the reaction rate is observed with the addition of lipase cloths. In difference,

214 when the concentration of tributyrin was increased to 99 mM (i.e. decreasing the catalyst: substrate ratio) a

215 significant positive effect can be observed for the 50cm catalyst cloth. The rate increased from 3.8 mM min^{-1}

216 (one cloth) to 6.96 mM min^{-1} (three cloths). At this higher substrate concentration, the reaction rate is limited

217 by the number of active sites present on the cloth. Hence, further showing that the reaction rate, and the

218 productivity can be tailored by increasing both the cloth size and cloth number at higher substrate

219 concentration.

220 Fig. 3(d) shows the increase in mean residence time with increasing cloth size and number. The mean

221 residence time for a 50 cm cloth is nearly twice that of three 20 cm cloths. Addition of cloths in the SMDR

222 leads to an increased resistance to the flow by the cloth fibre mesh creating multiple flow channels resulting

223 in a well-mixed reactor compared to a conventional spinning disc reactor which follows plug flow behaviour

224 [17].

225 These results indicate that the scale-up in the SMDR can be achieved by either increasing the cloth size or

226 cloth number. The increasing reaction rate with increasing cloth size is primarily due to the increase in catalyst

227 loading and the mean residence time. A further increase in the reaction rate can be achieved by numbering-up

228 the catalyst cloths of increasing diameters facilitating a tuneable operation of the SMDR.
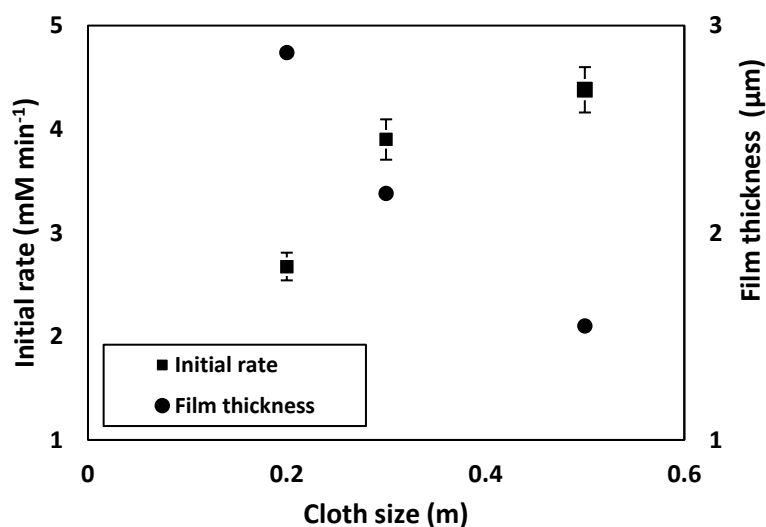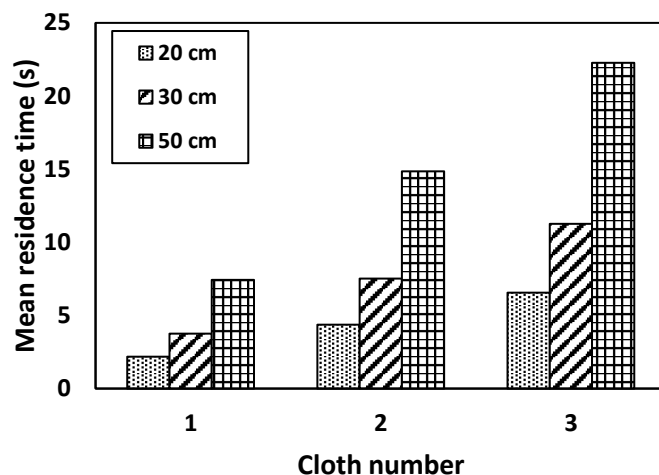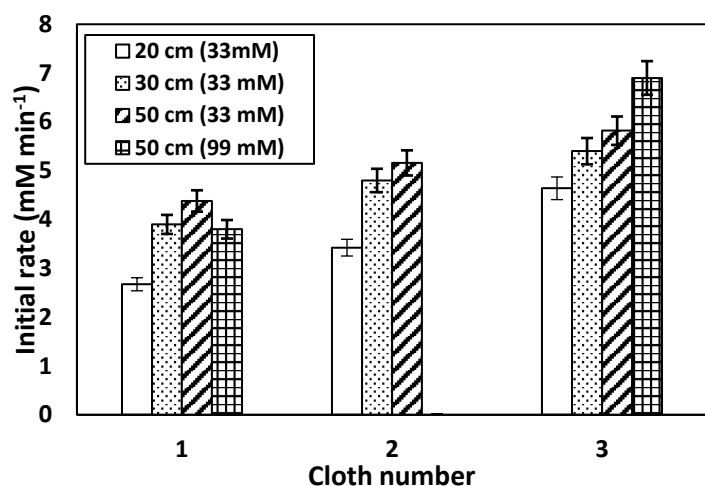
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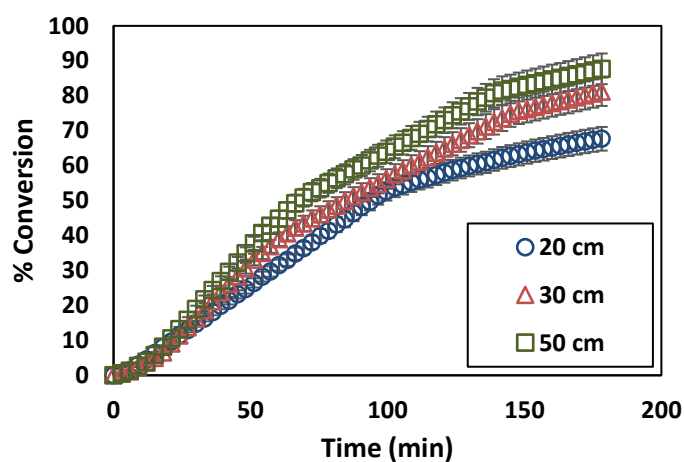
230

231   (a)

232

233   (b)

234

(c)   (d)

Figure 5: (a) Effect of cloth size (single cloth) on reaction conversion as a function of time, (b) Effect of

cloth size on initial reaction rate (substrate concentration 33 mM), (c) Comparison of initial reaction rates

for different cloth size and cloth numbers. The reactions were carried out at spinning speed of 350 RPM and

flowrate of 3 ml s^{-1} and (d) Mean residence time as a function of increasing cloth size and cloth number

3.2 Effect of changing cloth size and cloth number on nitroaldol condensation reaction in the SMDR

To establish the proof of concept for scale-up of organic reactions in the SMDR, nitroaldol condensation

reaction was carried out by altering the copper catalyst loading through increasing cloth size and cloth number.

Effect of cloth size: Fig 4(a) shows that the reaction conversion increased with increase in cloth size and the

reaction rate increased from 0.018 mmol min^{-1} (20 cm cloth) to 0.033 mmol min^{-1} (50 cm cloth). The overall

reaction rate co-efficient (k_{ovl}) increased from 5.86×10^{-6} m3/m$^2_{cloth}$ s^{-1} (20 cm cloth) to 1.40×10^{-5} m3/m$^2_{cloth}$ s^{-1} (50 cm cloth) indicating an increase in mass transfer from liquid to the cloth surface with increase in cloth

surface area. Further, the average surface shear with increasing cloth size and rotation speed for the organic

solvent system was nearly 1.5 times less compared to the aqueous system (supplementary information B). The

surface shear on the cloth surface is dependent on the fluid properties of the reaction medium, which varies

according to the solvent used and hence the increase in reaction rate for the solvent system is less profound

compared to the aqueous system. Hence, the increase in rate and productivity for organic reactions is mainly

252   due to the increase in the amount of catalyst immobilised with increasing cloth surface area and a longer

253   residence time on the cloth surface. The overall reaction rate co-efficient (k_{ovl})

254   **Effect of numbering-up cloths:** Similar to tributyrin hydrolysis, addition of multiple 20 cm cloths (up to 3

255   cloths) resulted in a reaction rate similar to that obtained using a 50 cm cloth. The reaction scale-up through

256   increasing cloth size and cloth number demonstrated a compounding effect on the reaction rate and the

257   maximum reaction rate of 0.043 mmol min^{-1} was obtained for three 50 cm cloths. Although the liquid feed

258   had penetrated through the entire cloth stack, a compounding effect in the reaction was not observed as seen

259   for the aqueous system at a higher substrate concentration. This is due to a lower volume of liquid entrapped

260   in the cloth stack (due to lower wettability of woollen cloth by ethanol compared to water) and hence lesser

261   contact with the feed stream and the catalyst.

262   Overall, the results indicate that the SMDR can be scaled for organic reactions either by increasing the cloth

263   size or cloth number as demonstrated for the above reaction. To the best of the authors' knowledge, this is the

264   first report on the scale-up of nitroaldol condensation reaction in the SMDR.
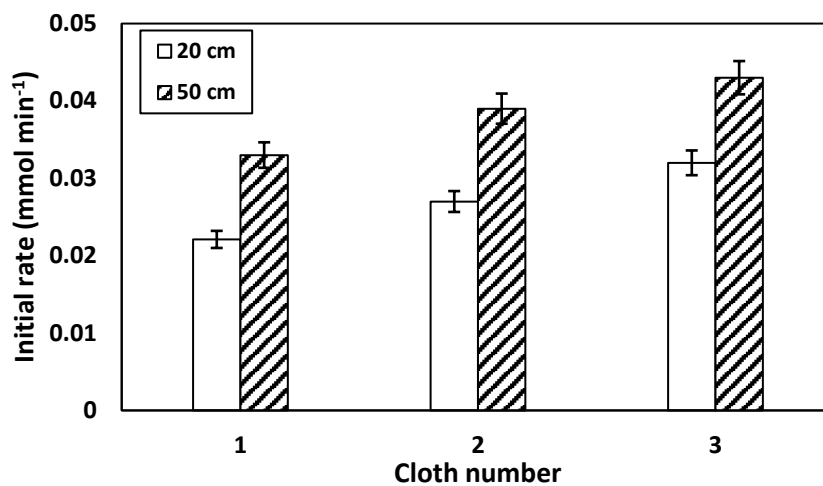
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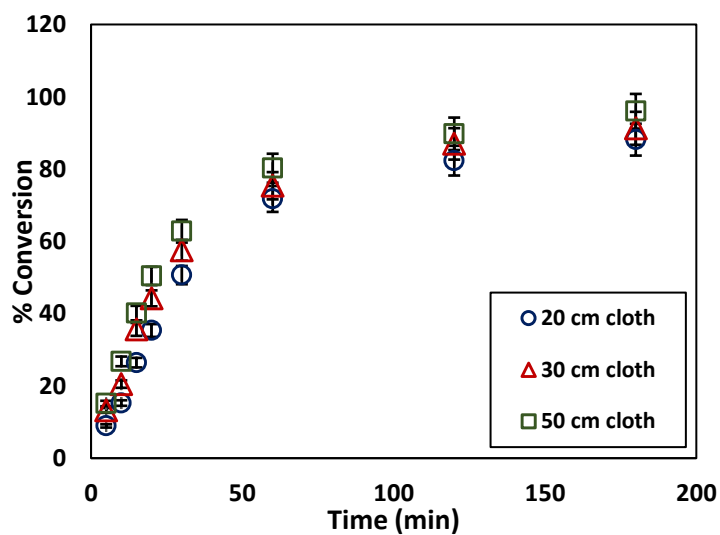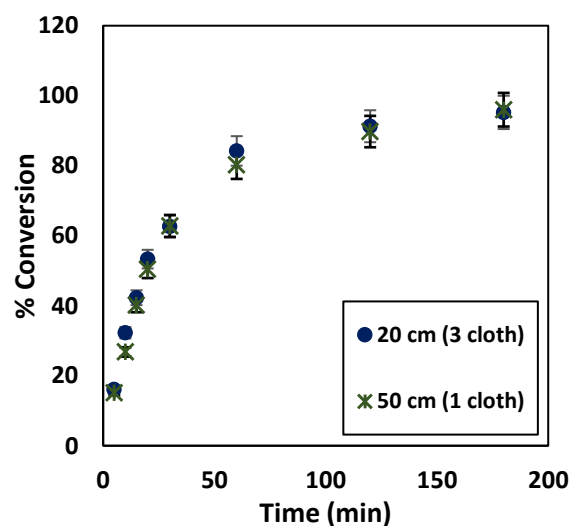
266   (a)   (b)

(c)

Figure 6: Nitroaldol condensation reaction (a) Effect of cloth size on reaction conversion as a function of time, (b) comparison between reaction conversion for three 20 cm cloths and a 50 cm cloth, (c) reaction rate comparison for scale-up by increasing cloth diameter vs increasing cloth number. All reactions were carried out at a spinning speed of 350 RPM and flowrate of 3 ml s^{-1}.

4. Conclusion

In this study, scale-up of SMDR was investigated using two routes: (i) increasing cloth size and (ii) addition of multiple cloths. The reactor performance was evaluated for the hydrolysis of tributyrin and nitroaldol condenstation reactions as the reactions were previously optimised in the SMDR. The reactions were carried out in a newly designed reactor allowing for various disc diameters and reduced spin-up zone on the disc surface. A two level, full factorial DoE study was carried out to understand the effect of spinning speed, cloth size and cloth number on the reaction rate. Results from the DoE studies indicated that increasing either cloth size or cloth number individually had a greater effect on the reaction rate for a given substrate concentration. Further, increasing the cloth size resulted in an increase in conversion and reaction rate as a result of increased surface area of the catalyst cloth. The reaction rate achieved with a single 50 cm cloth (4.38 mM min^{-1}) was comparable to that of three 20 cm cloths (4.64 mM min^{-1}) for a tributyrin substrate concentration of 33 mM. Addition of multiple cloths of increasing cloth size also showed an increase in the reaction rate and the reaction rate increased by 36% at a substrate concentration of 99mM for three 50 cm cloths. The study was extended to nitroaldol condensation reaction catalysed by copper immobilised on wool. Similar to the previous case, an increase in reaction conversion and rate was observed with increase in cloth size and cloth number and addition

288 of three 20 cm catalyst cloths yielded similar rate (0.032 mmol min^{-1}) to that of a single 50 cm cloth (0.033

289 mmol min^{-1}). Overall, it can be concluded that the reaction scale-up in the SMDR can be achieved by

290 increasing the cloth size and the cloth number for both aqueous and organic reaction systems, making it a

291 flexible reactor design for reaction intensification. Future work will aim to examine higher reaction throughput

292 for scale-up of organic reactions.

Acknowledgement

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