

# Peracetic Acid Synthesis by Acetaldehyde Liquid Phase Oxidation in Trickle Bed Reactor

ZHANG Tengyun(张腾云)<sup>a,\*</sup>, LUO Jingli(罗京莉)<sup>b</sup>, K. Chuang<sup>b</sup> and ZHONG Li(钟理)<sup>a</sup>

<sup>a</sup> College of Chemical and Energy Engineering, South China University of Technology, Guangzhou 510640, China

<sup>b</sup> Department of Chemical & Materials Engineering, University of Alberta, Edmonton, AB Canada, T6G 2G6

**Abstract** In this paper, shorter residence time (a few minutes) with high yield in the trickle bed process for peracetic acid synthesis by acetaldehyde liquid phase oxidation can be realized on the selected packing material SA-5118. For acetaldehyde in acetone with ferric ion as catalyst, the optimized process conditions were presented. The main factors influencing the yield, selectivity and conversion are residence time, temperature and acetaldehyde concentration, respectively. The temperature range checked is from 30 to 65 °C. High yield of 81.53% with high selectivity of 91.84% can be obtained at higher temperature of 55 °C when the residence time is 5.5 min and the acetaldehyde concentration is 9.85% (by mass). And there is a critical acetaldehyde concentration point ( $C_{cep}$ ) between 18% and 19.5% (by mass). At temperature less than 55 °C, the highest yield to peracetic acid at each temperature level increases with temperature when the acetaldehyde concentration is below  $C_{cep}$  and decreases with temperature when the acetaldehyde concentration is above  $C_{cep}$ .

**Keywords** peracetic acid, liquid phase oxidation, acetaldehyde, trickle bed

## 1 INTRODUCTION

During the past decade the chemical reactions of peroxides have been the subject of intensive study. Many reactions, with numerous oxidants, have been developed to bring about selective oxidation. These reactions resulted not only in new types of stable peroxides but also in unstable peroxides with potential application[1]. One of the most important organic peroxides is peracetic acid (PAA) which is cheaper compared with longer chain peroxy carboxylic acids, and more stable than peroxy formic acid. So PAA is a widely used and studied oxidizing agent in many industrial fields[2—9], which promotes the development of the process for PAA synthesis. The PAA synthesis processes that have been already commercialized can be classified into two types: the acetylation of hydrogen peroxide and the acetaldehyde oxidation process. There are three kinds of acetaldehyde oxidation processes: the two-step liquid phase oxidation, the vapor phase oxidation and the one-step liquid phase oxidation[10]. The acetylation process produces aqueous PAA product and only the acetaldehyde oxidation process produces nonaqueous PAA in organic solvent.

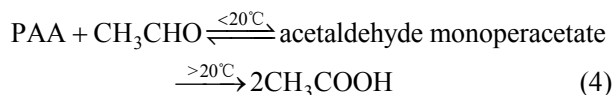
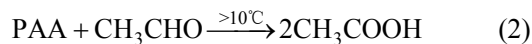
Because of the simpler process and the safer operation, the one-step liquid phase oxidation process, commercialized by Daicel of Japan in 1970[10], is more attractive than any of the other two acetaldehyde oxidation processes. The one-step liquid phase oxidation process uses tubular reactor with cocurrent upward flow operated at lower temperature of below 45 °C. The acetaldehyde conversion is still low (yield to PAA less than 50%), and at the higher conversions, the selectivity to PAA is greatly reduced[11]. With acidic catalyst[10,12], Although the acetaldehyde conversion are increased to 80%, the yield to PAA is just 70%. One of the main drawbacks using acidic catalyst is that the reaction rate is much slower compared with

that using metal catalyst. The one-step liquid phase oxidation process was also optimized by some researchers[13—15], but the yield to PAA is still below 75%. That the yield is difficult to increase in the one-step liquid phase oxidation is mainly because of the limitations of the old process itself: at low temperature, the reaction rate is slow and thus longer residence time is required, which will lead to much more decomposition of PAA and the yield decreases. And at high temperature, though the residence time is reduced, the side reactions to form byproducts also occur rapidly thus the yield can not be increased. So the solution for the demand for nonaqueous PAA through the one-step liquid phase oxidation is to reduce the high production cost and to get high PAA yield by increasing the conversion and selectivity and to increase PAA mass production capacity through the improvement on the old process.

In the one-step liquid phase oxidation process with metal ion ( $M^{n+}$ ) as the catalyst, the main reactions are as follows:



The important side reactions are[16]:



Theoretically, if completely plug flow state (without any backmixing) and full contact of acetaldehyde and oxygen can be realized, all acetaldehyde undergoes reaction (1) in shorter time, and 100% selectivity and yield to PAA can be achieved. However, the backmixing of PAA (once formed) with acetaldehyde in

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\* To whom correspondence should be addressed. E-mail: zty-265@tom.com

the liquid feed exists to some extent. Thus side reactions (2) and (4) can not be avoided. But side reaction (4) can be neglected when the temperature is higher than room temperature for the formation of acetaldehyde monoperoacetate (AMP) can be inhibited at higher temperature above 30°C [16]. Side reaction (3) is PAA decomposition. The shorter the residence time in the reactor, the less PAA decomposition occurs. The PAA decomposition rate also increases with the temperature and becomes quite rapid above 60°C [17]. So, to obtain high acetaldehyde conversion and PAA selectivity, higher temperature is required to increase the reaction rate and thus increase the acetaldehyde conversion and shorten the residence time. The formation of AMP is also inhibited. But the temperature can not be too high for inhibiting the PAA decomposition.

After 1980s, there seemed no development and improvement on the one-step liquid phase oxidation process. To improve the yield to PAA of the one-step liquid phase oxidation, the purpose of this study is to provide an innovated trickle bed process, which can provide a plug flow state in the reaction system. The new process uses packing material and trickle bed in the one-step liquid phase oxidation. The backmixing is reduced to the least compared with the countercurrent flow and cocurrent upward flow. Good packing material is selected to provide a good contact for oxygen and liquid acetaldehyde. Another purpose is to optimize the operation conditions to make the process economical and efficient for the large scale production of PAA in high yields.

## 2 EXPERIMENTAL

### 2.1 Synthesis process

The schematic diagram of PAA synthesis process is shown in Fig.1. Oxygen comes from an oxygen cylinder and its flow rate is controlled and indicated by a 8270 Matheson mass flow controller. The flow rate of the liquid feed containing acetaldehyde, acetone and ferric acetylacetonate as catalyst ( $0.4\text{mg}\cdot\text{kg}^{-1}$  ferric ion) from the liquid feed vessel is controlled by Waters 515 HPLC pump. The product is collected at

room temperature so that little AMP can be formed in the product. The absorber containing the solvent of xylene is cooled by ice and water so that most of the vapor acetaldehyde and acetone in the tail gas can be absorbed

### 2.2 Trickle bed reactor

The trickle bed reactor in Fig.2 is a stainless steel (316) tube with packing materials inside and a cooling jacket outside to adjust the reactor temperature. The length of the packing bed is 31cm and the inner diameter is 10mm. The nonporous packing material, SA-5118 (5mm×5mm Rasching rings, ceramic of  $\alpha$ -alumina.), from Saint-Gobain NorPro with rough surface is inert to strongly oxidative PAA and is crashed to 0.42—0.84mm (20—40 meshes) to provide good contacting area. It is the best packing material so far found for PAA synthesis. A Teflon-coated thermocouple is inserted into the middle of the packing bed from the bottom of the reactor to record the temperature fluctuation. Glass wool is also put at the bottom to prevent small packing material particles from clogging the reactor outlet at the bottom. A small tube for liquid feed to enter the reactor is inserted at the top of the reactor, and the oxygen inlet is also at the top of the reactor.

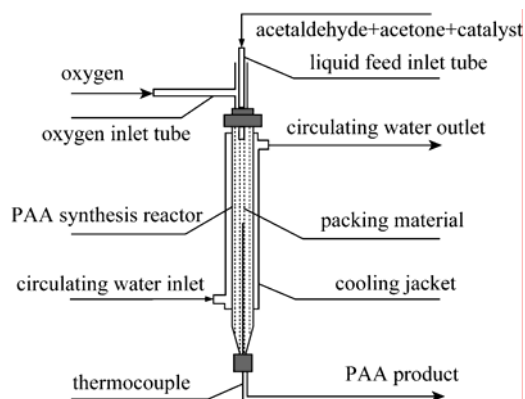


Figure 2 Trickle bed reactor for PAA synthesis

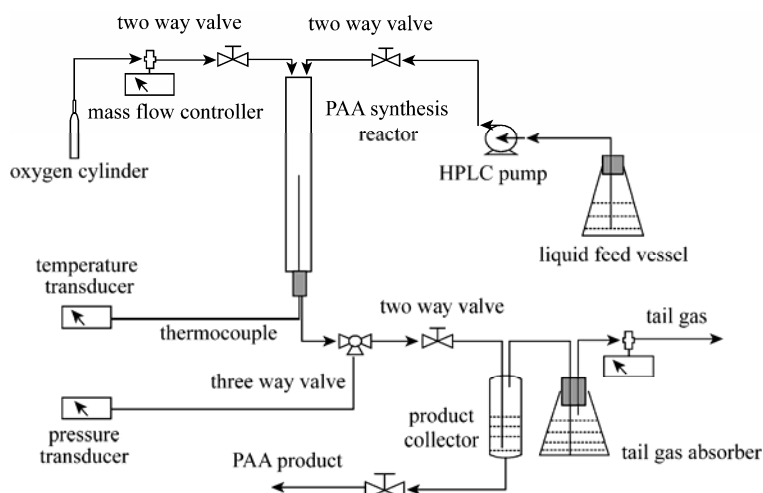


Figure 1 PAA synthesis process

### 2.3 Product analysis

All chemicals used were of reagent grade. The PAA in the product was analyzed by titration[18]. No AMP was detected. Other components in the product, liquid feed, absorber solvent and tail gas were analyzed by gas chromatography (Agilent Technologies 6890N) with thermal conductivity detector (TCD). Liquid sample was injected with a syringe and the tail gas is injected via a built-in six-way valve in the gas chromatography (GC).

## 3 RESULTS AND DISCUSSION

### 3.1 Packing materials selection

For gas-liquid reaction of PAA synthesis by acetaldehyde oxidation, the packing material not only provides the contacting area but also influences the residence time. Large volume of inner pores in the packing material will increase the residence time and backmixing of PAA with acetaldehyde. So, highly porous and very smooth packing materials are inappropriate. This conclusion is conformed by the PAA synthesis results using the following packing materials: SA-5118 (US ceramic,  $\alpha$ -alumina, from Saint-Gobain NorPro, USA); F-ceramic ( $\alpha$ -alumina, from Mallinckrodt Baker, Canada); alumina ( $\gamma$ - $\text{Al}_2\text{O}_3$ , from Mallinckrodt Baker, Canada); glass pellets (from Aldrich); silica-alumina ( $\text{SiO}_2$  53%;  $\text{Al}_2\text{O}_3$  45%, from Aldrich). The physical properties and surface morphology of the packing materials are shown in Fig.3 and Table 1, respectively.

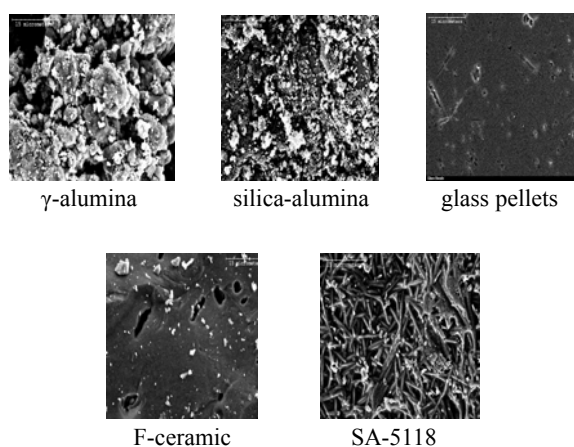


Figure 3 SEM graphs of packing materials (magnified 2000 times)

From Fig.3 and Table 1,  $\gamma$ -alumina and silica-alumina are porous with large surface area, and large porous area provides poor selectivity to PAA. Another reason may be that they are active material in contrast with ceramic and glass, for PAA is strongly oxidative which is instable to active materials. Glass pellet has very small area and thus provides the lowest conversion, but the smooth area gives good selectivity. SA-5118 having the appropriate surface area and nonporous surface can provide the best conversion, selectivity and yield. In the subsequent experiment, SA-5118 is selected as the packing material and pure oxygen as the gas.

### 3.2 Optimization of PAA synthesis conditions

Previous work showed that acetone is the best solvent for PAA synthesis and a little amount of ferric ion as catalyst ( $0.2\text{mg}\cdot\text{kg}^{-1}$ ) is good enough[17]. So for acetaldehyde in acetone as liquid feed with ferric ion as catalyst ( $0.4\text{mg}\cdot\text{kg}^{-1}$ ) to synthesize PAA by one-step liquid phase oxidation, when the reaction system (reactor configuration and packing material) is determined, the yield ( $Y$ ) and selectivity ( $S$ ) to PAA depend on the integrated effect of the following factors: pure oxygen pressure ( $p$ ), pure oxygen flow rate ( $G$ ), temperature ( $T$ ), residence time ( $t$ ) (adjusted by liquid feed flow rate,  $F$ ); acetaldehyde mass concentration ( $C$ ).

It is also confirmed that the oxygen partial pressure has little influence on the PAA synthesis when  $p > 1.379 \times 10^5$  Pa (oxygen partial pressure). So the influence of oxygen partial pressure on the conversion ( $X$ ), selectivity and yield can be neglected when  $p$  is appropriate.

#### 3.2.1 Effect of oxygen flow rate

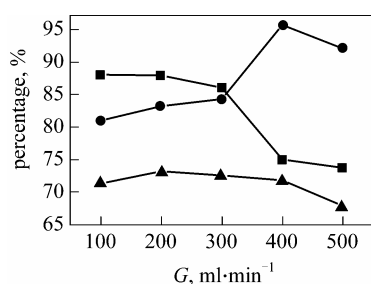
Figure 4 shows that the conversion  $X$  decreases with oxygen flow rate  $G$  and decreases sharply when  $G$  is higher than  $300\text{ml}\cdot\text{min}^{-1}$ , because high oxygen flow rate reduces the residence time of liquid feed in the reactor. The selectivity and yield increase with  $G$  at lower oxygen flow rate and decrease with  $G$  when oxygen flow rate is higher than  $500\text{ml}\cdot\text{min}^{-1}$ . The highest yield is obtained at around  $200\text{ml}\cdot\text{min}^{-1}$ . Considering the conversion, selectivity and yield, the oxygen flow rate should be from  $200$  to  $300\text{ml}\cdot\text{min}^{-1}$ .

Another reason limiting the oxygen flow rate is that the loss of acetaldehyde and acetone in the tail gas increases sharply with oxygen flow rate as shown in Fig.5. When  $G$  is below  $300\text{ml}\cdot\text{min}^{-1}$ , the solvent loss is less than  $0.75\%$  which can be neglected.

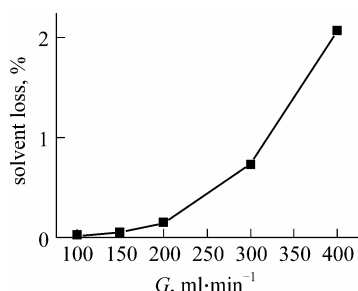
Table 1 Surface properties and PAA synthesis results of packing materials

Packing material	Surface area	Surface morphology	$C$ , %	$S$ , %	$Y$ , %
$\gamma$ -alumina	very large	high porous	61.16	0	0
silica-alumina	large	porous	60.34	7.2	4.34
F-ceramic	small	porous to smooth	85.3	64.38	54.92
glass pellets	very small	very smooth	52.2	83.94	43.84
SA-5118	medium	rough and non porous	89.15	86.87	77.44

Note: All the materials are crashed to  $0.42\text{--}0.84\text{mm}$  ( $20\text{--}40$  mesh) and the sample of each run is taken after 20min.  $G=150\text{ml}\cdot\text{min}^{-1}$ ,  $t=8.23\text{min}$ ,  $F=1.0\text{ml}\cdot\text{min}^{-1}$ ,  $T=55^\circ\text{C}$ ,  $p=7.585 \times 10^5\text{N}\cdot\text{m}^{-2}$ ,  $C=10\%$ ,  $0.4\text{mg}\cdot\text{kg}^{-1}\text{Fe}^{3+}$ , acetone as the solvent, pure oxygen as the gas.



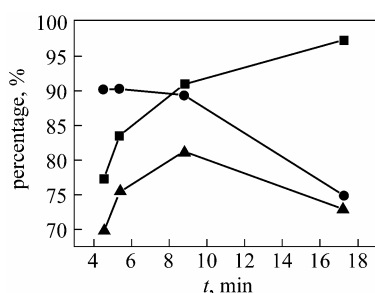
**Figure 4** Effect of  $G$  on reaction ( $T=55^{\circ}\text{C}$ ,  $t=4.25\text{min}$ ,  $F=2.0\text{ml}\cdot\text{min}^{-1}$ ,  $C=9.81\%$ ,  $p=7.791\times 10^5\text{Pa}$ )  
 ■ X; ● S; ▲ Y



**Figure 5** Influence of  $G$  on solvent loss ( $T=55^{\circ}\text{C}$ ,  $t=8.17\text{min}$ ,  $F=1.0\text{ml}\cdot\text{min}^{-1}$ ,  $C=10.39\%$ ,  $p=5.585\times 10^5\text{Pa}$ )

### 3.2.2 Effect of residence time

Residence time  $t$  of acetaldehyde in the reactor has great influence on the reaction results. Low liquid feed flow rate means long residence time for the reactant in the reactor, leading to higher conversion. Longer residence time will also lead to more decomposition of PAA, so the PAA yield and selectivity will be decreased. But too short residence time (or high  $F$ ) will also lead to low conversion of acetaldehyde and low PAA yield. If the acetaldehyde conversion is too low, more residue acetaldehyde will lead to more side reaction (2) and low PAA selectivity. So there is a proper residence time for the highest selectivity and yield. From Fig.6, the highest yield ( $Y_{\text{MTC}}$ ) and selectivity ( $S_{\text{MTC}}$ ) (obtained from different residence time at certain temperature and certain acetaldehyde concentration) for  $T=50^{\circ}\text{C}$ ,  $C=9.85\%$  are 81.23% and 90.23%, respectively. And the corresponding residence time  $t_Y$  and  $t_S$  for  $Y_{\text{MTC}}$  and  $S_{\text{MTC}}$  are  $t_Y=8.78\text{min}$  and  $t_S=5.37\text{min}$ , respectively.

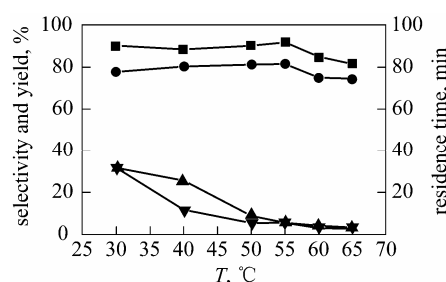


**Figure 6** Effect of residence time on reaction ( $T=50^{\circ}\text{C}$ ,  $C=9.85\%$ ,  $p=8.136\times 10^5\text{Pa}$ ,  $G=200\text{ml}\cdot\text{min}^{-1}$ )  
 ■ X; ● S; ▲ Y

### 3.2.3 Effect of temperature

Theoretically, the reaction rate increases with temperature and side reaction (2) is inhibited. It seems that the selectivity and yield to PAA would increase with temperature. However, it is not the case because of the rapid side reaction (3) caused by increasing temperature. So there is an appropriate temperature for obtaining the highest yield and selectivity in PAA synthesis. As described above, in the proper range of oxygen flow rate  $G$ , parameters  $t$ ,  $T$  and  $C$  will influence the values of  $Y$  and  $S$ .

Then for certain  $C$  at each  $T$ , with the results obtained in Section 3.2.2, there are  $Y_{\text{MTC}}$  and  $S_{\text{MTC}}$ . The influence of  $T$  on  $Y_{\text{MTC}}$  and  $S_{\text{MTC}}$  at different temperature is shown in Fig.7.



**Figure 7** Effect of temperature on the  $Y_{\text{MTC}}$ ,  $S_{\text{MTC}}$  and corresponding residence time ( $C=9.85\%$ ,  $p=8.136\times 10^5\text{Pa}$ ,  $G=200\text{ml}\cdot\text{min}^{-1}$ )  
 ■  $S_{\text{MTC}}$ ; ●  $Y_{\text{MTC}}$ ; ▲  $t_Y$ ; ▼  $t_S$

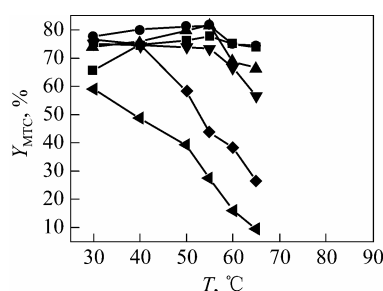
For  $C=9.85\%$ , from Fig.7, the highest yield ( $Y_{\text{MTC}}$ ) at each temperature increases slightly with temperature below  $55^{\circ}\text{C}$  and decreases when temperature is above  $60^{\circ}\text{C}$ . And the maximum value of  $Y_{\text{MTC}}$  and  $S_{\text{MTC}}$  (defined as  $Y_{\text{MC}}$  and  $S_{\text{MC}}$  respectively) for  $C=9.85\%$  at different temperature and residence time are 91.84% and 81.53%, respectively. The corresponding residence time  $t_Y$  and  $t_S$  for  $Y_{\text{MC}}$  and  $S_{\text{MC}}$  are the same:  $t_Y=t_S=5.50\text{min}$  and the corresponding temperature is  $T=55^{\circ}\text{C}$ .

It can be also found that the residence time for  $Y_{\text{MTC}}$  and  $S_{\text{MTC}}$  greatly depends on the temperature. At low temperature of  $30^{\circ}\text{C}$ ,  $t_Y$  and  $t_S$  are all 31.58min, which is very long compared with the residence time at  $55^{\circ}\text{C}$  ( $t_Y=t_S=5.50\text{min}$ ). The results also show that with the trickle bed process and the selected packing material, the maximum yield and selectivity can be achieved within a few minutes at higher temperature, which is attributed to the inhibition of side reaction (3) by shortening the residence time.

### 3.2.4 Effect of acetaldehyde concentration on yield to PAA

As stated above, For  $C=9.85\%$ , the highest yield,  $Y_{\text{MC}}=81.53\%$ , can be obtained at  $T=55^{\circ}\text{C}$  and  $t=5.50\text{min}$  and the corresponding  $S_{\text{MC}}=91.84\%$ . For different  $C$ , the  $Y_{\text{MC}}$  can be obtained through the change of  $Y_{\text{MTC}}$  with different temperature using the same method described above.  $Y_{\text{MTC}}$  of different temperature for different  $C$  is shown in Fig.8.

From Fig.8, for acetaldehyde concentration less than 16.04% (by mass), the  $Y_{\text{MTC}}$  increases with temperature below  $55^{\circ}\text{C}$  and decreases with temperature

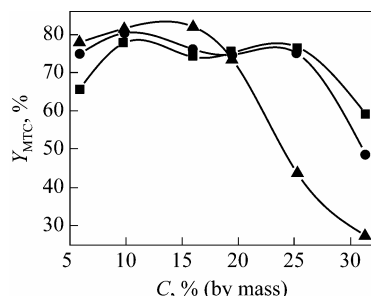


**Figure 8** Change of the yield  $Y_{MTC}$  with temperature for different acetaldehyde concentration ( $p=8.136 \times 10^5$  Pa,  $G=200$  ml·min $^{-1}$ )  
 $C$ , %: ■ 5.87; ● 9.85; ▲ 16.04; ▼ 19.46; ◆ 25.26; ◀ 31.35

above 60°C, and there is a maximum value of  $Y_{MTC}$  (defined as  $Y_{MC}$ ) at 55°C.  $Y_{MC}$  for  $C$  of 5.87%, 9.85% and 16.04% is 77.88%, 81.53% and 81.90%, respectively, and the corresponding residence time  $t_Y$  is 5.47min, 5.50min and 4.63min respectively. But for acetaldehyde concentration more than 19.46% (by mass), in the temperature range from 30 to 65°C, there is no maximum value of  $Y_{MTC}$  and the  $Y_{MTC}$  decreases with temperature all the time.

It is obvious that there is a critical acetaldehyde concentration point (defined as  $C_{ccp}$ ) between 16.04% (by mass) and 19.46% (by mass), at temperature below 55°C, the yield  $Y_{MTC}$  increases with temperature when  $C < C_{ccp}$  and decreases with temperature when  $C > C_{ccp}$ . The reason why there is a  $C_{ccp}$  is that for low acetaldehyde concentration, the main reaction is reaction (1) and most acetaldehyde can be reacted. The selectivity depends mainly on side reaction (3). With temperature increasing, the reaction rate increases, high conversion can be obtained within shorter residence time and thus the side reaction (3) is inhibited. So the yield is higher. For high acetaldehyde concentration, there is excessive acetaldehyde in the liquid feed. With temperature increasing the side reaction (2) will be dominant and the yield to PAA will decrease.

$C_{ccp}$  can also be clearly shown in Fig.9, where  $Y_{MTC}$  changing with acetaldehyde concentration for certain temperature is plotted. From Fig.9,  $C_{ccp}$  can be more accurately determined between 18% (by mass) and 19.5% (by mass).



**Figure 9** Change of the yield  $Y_{MTC}$  with acetaldehyde concentration for different temperature ( $p=8.136 \times 10^5$  Pa,  $G=200$  ml·min $^{-1}$ )  
 ■ 30°C; ● 40°C; ▲ 55°C

From all discussions above, the optimized reaction conditions to get high yield for PAA synthesis in the process described in this paper are as follows:  $p > 1.379 \times 10^5$  Pa,  $G=200-300$  ml·min $^{-1}$ ,  $T=55$  °C,  $t=4.5-6.0$  min,  $C < 18\%$ .

## 4 CONCLUSIONS

The trickle bed process for peracetic acid synthesis is an innovated process not reported before, which can shorten the residence time to a few minutes and reduce the backmixing to the least thus to inhibit the side reactions to obtain high yield to peracetic acid. High yield of 81.53% with high selectivity of 91.84% for PAA synthesis can be obtained in this process under the proper conditions. The detailed discussion of the PAA synthesis process conditions is a very useful reference to large scale production. The results about the influence of acetaldehyde concentration in liquid feed for the trickle bed process indicate that there is a critical point of acetaldehyde concentration which should be taken seriously for PAA synthesis.

## NOMENCLATURE

$C$	acetaldehyde mass concentration in liquid feed, %
$F$	liquid feed flow rate, ml·min $^{-1}$
$G$	pure oxygen flow rate, ml·min $^{-1}$
$p$	oxygen partial pressure at the outlet of reactor, Pa
$S$	selectivity of peracetic acid, %
$S_{MTC}$	highest selectivity of peracetic acid, %
$S_{MC}$	maximum $S_{MTC}$ , %
$T$	temperature in the middle of reactor, °C
$t$	residence time, min
$t_s$	residence time for corresponding selectivity, min
$t_Y$	residence time for corresponding yield, min
$X$	conversion of acetaldehyde, %
$Y$	yield to peracetic acid, %
$Y_{MTC}$	highest yield to peracetic acid (by mass), %
$Y_{MC}$	maximum $Y_{MTC}$ , %

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