

การออกแบบและการควบคุมหอกลับแบบเกิดปฏิกิริยา  
สำหรับการผลิตบิวทิลอะซิเตตจากกรดอะซิติกเจือจาง



นางสาวจันทรวดี วิวิธนาภรณ์

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต

สาขาวิชาวิศวกรรมเคมี ภาควิชาวิศวกรรมเคมี

คณะวิศวกรรมศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

ปีการศึกษา 2549

ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

DESIGN AND CONTROL OF A REACTIVE DISTILLATION COLUMN  
FOR THE PRODUCTION OF BUTYL ACETATE FROM DILUTE ACETIC ACID

Miss Chantarawadee Wiwittanaporn

A Thesis Submitted in Partial Fulfillment of the Requirements  
for the Degree of Master of Engineering Program in Chemical Engineering

Department of Chemical Engineering

Faculty of Engineering

Chulalongkorn University

Academic Year 2006

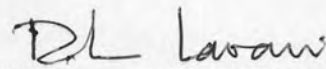
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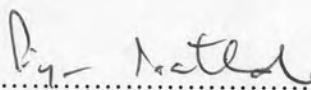
Thesis Title                      DESIGN AND CONTROL OF A REACTIVE  
DISTILLATION COLUMN FOR THE PRODUCTION OF  
BUTYL ACETATE FROM DILUTE ACETIC ACID  
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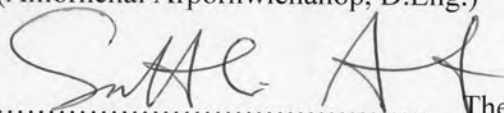
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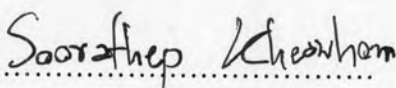
  
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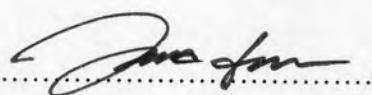
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การนำกรดอะซิติกเจือจางกลับไปใช้ประโยชน์เป็นเรื่องที่มีความสำคัญเพื่อเป็นการเพิ่มมูลค่าทางเศรษฐศาสตร์แล้วยังเป็นการลดปัญหาทางสิ่งแวดล้อมอีกด้วย งานวิจัยนี้มุ่งเน้นที่การประยุกต์ใช้การกลั่นแบบมีปฏิกิริยาสำหรับนำกรดอะซิติกเจือจางกลับไปใช้ประโยชน์ โดยศึกษาการสังเคราะห์บิวทิลอะซิเตดจากปฏิกิริยาเอสเทอร์ฟิเคชันของกรดอะซิติกเจือจางและบิวทานอลในหอกลั่นแบบมีปฏิกิริยาโดยใช้โปรแกรมไฮซิส (HYSYS) ผลการคำนวณที่ได้จากแบบจำลองโดยใช้โปรแกรมไฮซิสถูกเปรียบเทียบกับผลการทดลองที่รายงานในวารสารวิชาการ จากการเปรียบเทียบพบว่าผลการคำนวณมีความถูกต้อง หลังจากนั้นได้เริ่มศึกษาถึงผลของความเข้มข้นของกรดอะซิติกเจือจางที่มีต่อการผลิตบิวทิลอะซิเตด นอกจากนี้ยังได้ศึกษาผลของตัวแปรการดำเนินงานที่สำคัญเพื่อใช้ในการออกแบบหอกลั่นแบบมีปฏิกิริยา สุดท้ายได้ทำศึกษาการควบคุมแบบวงปิดของหอกลั่นแบบมีปฏิกิริยาโดยได้ทำการออกแบบโครงสร้างการควบคุม 2 โครงสร้างสำหรับการผลิตบิวทิลอะซิเตดโดยใช้กรดอะซิติกที่มีความเข้มข้น 80% โดยน้ำหนัก และได้ทำการทดสอบระบบควบคุมโดยกำหนดให้อัตราการไหลและความเข้มข้นของกรดอะซิติกที่เข้าหอกลั่นมีการเปลี่ยนแปลง

ภาควิชา ..... วิศวกรรมเคมี  
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ลายมือชื่อนิสิต ..... จันทรวดี วิวิธนาภรณ์  
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ลายมือชื่ออาจารย์ที่ปรึกษาร่วม.....



## 4870244121 MAJOR CHEMICAL ENGINEERING

KEY WORDS: RECOVERY/ DILUTE ACETIC ACID/ REACTIVE DISTILLATION/  
BUTYL ACETATE PRODUCTION

CHANTARAWADEE WIWITTANAPORN: DESIGN AND CONTROL OF A  
REACTIVE DISTILLATION COLUMN FOR THE PRODUCTION OF BUTYL  
ACETATE FROM DILUTE ACETIC ACID. THESIS ADVISOR: AMORNCHAI  
ARPORNWICHANOP, D.Eng., THESIS COADVISOR: ASSOC. PROF.  
SUTTICHA ASSUBUMRUNGRAT, Ph.D., 86 pp.

Recovery of the dilute acetic acid becomes important issues due to economic and environmental awareness. The present research is focused on the application of a reactive distillation as a potential alternative method for dilute acetic acid recovery. The synthesis of butyl acetate, from esterification of dilute acetic acid with butanol in the reactive distillation is investigated with HYSYS commercial software. The model prediction by the HYSYS simulator is validated with experimental results reported in literature and a good agreement is observed. The effect of dilute acetic acid concentration on reactive distillation performances in terms of butyl acetate production is first studied. The influences of key operating variables on the performance of the reactive distillation are also evaluated in order to design the reactive distillation. Finally, the closed-loop control of a reactive distillation column is explored. Two alternative control structures are evaluated using 80 wt% acetic acid concentration for the production of butyl acetate. Disturbances in fresh feed flow rate and compositions are made to examine the rangeability of the control structures.

Department ..... Chemical Engineering  
Field of study ..... Chemical Engineering  
Academic year ..... 2006

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## ACKNOWLEDGEMENTS

The author would like to thank and express her sincere gratitude to her advisor, Dr. Amornchai Apornwichanop, and co-advisor, Associate Professor Suttichai Assabumrungrat, for their valuable suggestions, useful discussions throughout this research. Moreover, the author would also be thankful to Professor Piyasan Prasertdam, as the chairman, and Dr. Soorathep Kheawhom and Assistant Professor Worapon Kiatkittipong, as the members of the thesis committee for many valuable suggestions.

The financial supports from the Graduate school of Chulalongkorn University are gratefully acknowledged. The author also thanks to the members of the Control and Systems Engineering Laboratory for their assistance.

Finally, the author would like to express the highest gratitude to her parent and family for their love, inspiration, encouragement and financial support throughout this study.

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## NOMENCLATURE

$a_i$	activity of component $i$	[-]
$A_{ij}$	binary parameters of NRTL equation	[-]
$B_{ij}$	binary parameters of NRTL equation	[-]
$G_{ji}$	binary interaction parameter in NRTL equation	[-]
$K_c$	controller gain	
$k_f$	the forward rate	[kg/m <sup>3</sup> · s]
$K_{eq}$	the reaction equilibrium constants	[-]
$n$	total number of components	[-]
Nr	number of rectification zone	[tray]
Nrxn	number of reactive zone	[tray]
Ns	number of stripping zone	[tray]
$P$	pressure	[atm]
$\Delta P$	pressure drop	[atm]
RR	reflux ratio	[-]
$T$	temperature	[K]
$V_{\max}$	maximum velocity	[ft/sec]
$x_i$	mole fraction of component $i$	[-]
$X_i$	conversion of component $i$	[%]

### Abbreviations

BuOAc	butyl acetate	[-]
BuOH	butanol	[-]
H <sub>2</sub> O	water	[-]
HOAc	acetic acid	[-]

**Greeks Symbols**

$\alpha_{ij}$	relative volatility of component $i$ with respect to component $j$ for VLE ( $\alpha_{ij} = \alpha_{ji}$ )	[-]
$\gamma_i$	activity coefficient of component $i$	[-]
$\rho_V$	vapor density	[lb/ft <sup>3</sup> ]
$\tau_{ij}$	binary interaction parameter	[-]
$\tau_I$	integral time	[min]
$\tau_d$	derivative time	[min]