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REACTIVE SEPARATION: AN ALTERNATE PROCESS TECHNOLOGY (REVIEW)

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Abstract

Chemical Industries are the primarily, intended for conversion of raw materials into desired products that we use in our daily life. The chemical industry is undergoing huge changes worldwide. The challenge for chemical industries is to cut their costs while ensuring that they conform to the best practices in protecting the environment. Currently, chemical production is a high-tech industry, where the competitiveness is more based on investment on research and development than the labor cost. Reactive separation is part of process intensification, desired for operating cost and investment cost saving and scale up for industrial process. Combine reaction and physical separation in to single operation is the main aim of reactive separation. It is not new concept but researcher develops the interest in recent years as part of economic aspects. Also it reflects increase the literature in development of reactive separations. This review covers the work in reactive separation field

Keywords:

Reactive separation ;
Reactive membrane;
Reactive crystallization;
Reactive adsorption;
Reactive Distillation.

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1. Introduction

Chemical engineering industries perform the basic task of converting raw materials into useful products, which involves reaction and separation. The most complex step, in many instances, is the reaction itself. It may be tedious, time consuming or costly or in some cases all of these. It is obvious that any attempt to improve the reaction step will help that particular industry. Converting batch process into continuous one, reducing the process steps and employing techniques like process intensification are some ways to achieve this. Process intensification carried out by using modification in method of production or modification in equipment. Reactive separation is the one of component in process intensification.

Reactive separations are operations that combine the reaction and separation into a single unit and production and removal of products. Reactive separation covered cost saving for investment and operating cost. This technique was used in industry from six decades. Some of advantages of reactive separation are by using reaction to improve separation while others are realized by using separation to improve reactions like increase overall rate of reaction, overcoming equilibrium limitations. There are some disadvantages like, operating conditions of reaction and separation, modeling of process, scale up issue and higher development cost.

Reactive separations are useful to reactions, such as esterification, trans-esterification etherification, hydrolysis and alkylation. There are different reactive separation process such as reactive extraction (RE), reactive distillation (RD) and reactive absorption (RAB), reactive membrane separation (RMS), reactive crystallization (RC), reactive adsorption (RAD). Organization of the article thus: section 1 introduces to reaction separation. Section 2 describes the reactive extraction. Section 3 describes the reactive distillation, section 4 reactive absorption, section 5 reactive membrane separation section 6 reactive adsorption section 7 reactive crystallsation and the paper is concluded in section 8.

2. 2. Reactive extraction

Reactive extraction is carried out by contacting the aqueous phase with organic phase containing suitable extractant. After the extraction organic phase is collected and organic acid are recovered by distillation .The reactive extraction used for biodiesel production [1] and also acid and phenols are extracted by different types of amines and ammonium salt [2].

Aliphatic amines and phosphoric solvents are proposed as effective extractants by earlier researchers. [3]. Nonaromatic, water immiscible and polar solvents are preferred for the extraction. [4]. This separation method has advantages such as effective at high concentration of substrate in the extractive fermentation, enhanced recovery of acid, minimization downstream process. Several researchers worked on reactive extraction process. The details given in Table 1

Table 1 A summery sheet of reactive extraction processes

Purpose	**Process conditions**	**Comments**	**Reference**
Recovery of Carboxylic Acids.	tertiary aliphatic amines as extractants.	Studies involve fermentation with reactive extraction which offers several advantages, such as a high product yield and theremoval of byproducts.	[5]
RE of Gallic Acid.	tri-n-caprylylamine as extractants , temperature 278 K.	Studied a three level full factorial central composite design for process parameters on the reactive extraction of gallic acid from aqueous phase with tri-ncaprylylamine in hexanol.	[6]
RE of aldehydes from aqueous solutions.	Primene JM-T extractan- ts, temperature 278 K.	The reactive extraction of aldehydes with primary aminePrimene JM-T was explored. equilibrium model using physical solubility and chemical reaction developed.	[7]
RE of carboxylic acids using organic solvents andsupercritical fluids.	1-octanol and supercar- itical CO_2 as extractant, temperature 288 - 338 K.	Study showed that supercritical reactive extraction process is an efficient method of carboxylic acid separation from aqueous solutions. This method was simple and gives high yield.	[8]

RE of propionic acid.	extractants (tri-n-butylphosphate (TBP), tri-n-octylamine (TOA) and Aliquat 336).	Sunflower oil is a non-toxic diluent used in study. Extraction of propionic acid using different extractants like TBP, TOA and Aliquat 336 in sunflower oil was carried out. quaternary amine Aliquat 336 is the best extractant was found.	[9]
RE of Pyruvic Acid.	tri-n-octylamine as extractant, temperature 303K.	Studied reactive extraction of pyruvic acid using tri-n-octylamine as an extractant dissolved in an active (decanol) and in inert (kerosene) diluents, which decrease purification cost.	[10]
Reactive Extraction of Alcohols.	Temperature 333 - 423 K, cyclic anhydrides was used.	Modification of Aromatic, cyclo-aliphatic, and linear aliphatic alcohols can be achieved efficiently in the apolar organic solvent without using catalyst. Conversion higher than 90% can be achieved using 3 - methyl Gultaric anhydride.	[11]
RE of Citric Acid.	Extractants like tri-n-butylphosphate (TBP), tri-n-octylamine (TOA),and Aliquat 336 (A336) used , temperature 300 K.	Studied the reactive extraction equilibria of citric acid along with LSER modeling and process optimization. TOA most efficient extractant for the recovery of citric acid.	[12]
RE of Oxoethanoic Acid.	Amberlite-LA2 used as extractant, temperature 298 K.	Studied Amberlite-LA 2 using diluents from different for the reactive extraction of oxoethanoic acid. High Efficiency was found for Amberlite - LA2 andhexane-2-one diluent system.	[13]

3 Reactive Distillations

Reactive Distillation (RD) is a process in which reaction and distillation in a single vessel. It is gaining importance for the synthesis of reversible or consecutive chemical reactions for exceeding the equilibrium conversion and reducing the rate of formation of byproducts. These products must be removed from the column, by distillation, to increase the efficiency of the system [14, 15]. Improved selectivity, increased conversion, better heat control, effective utilization of reaction heat, scope for difficult separations and the avoidance of azeotropes are a few of the advantages that are offered by RD. The introduction of an in situ separation process in the reaction zone or vice versa leads to complex interactions between vapor–liquid equilibrium, mass transfer rates, diffusion and chemical kinetics, which poses a great challenge for design and synthesis of these systems. The use of RD for every reaction may not be feasible. Exploring the candidate reactions for RD, it is an area that needs considerable attention to expand the domain of RD processes. There several constraints for reactive distillation like volatility constraints, residence time requirement, Scale up to large flows, Process conditions mismatch. [16]. Besides these constraintsStadig[17] showed that the RD become interesting options for reactions like Decomposition of ethers to high purity olefins, Dimerization, Alkylation of aromatics and aliphatics, esterifications, Hydroisomerizations, Hydrolyses, Dehydrations of ethers to alcohols, Oxidative dehydrogenations, Carbonylations, C_1 chemistry reactions, e.g. methyl from formaldehyde and methanol. Work done of various researchers in RD is given in Table 2.

Table 2. A summary sheet of reactive distillation processes

Purpose	**Process conditions**	**Comments**	**Reference**
Control study of production of methyl acetate.	temperature 350 –430 K and at a pressure of 1 atm.	Investigated control of a methyl acetate reactive distillation column. Several steady-state multiplicities are found. The effect of these multiplicities on the open loop stabilities was discussed.	[18]
Design alternatives for the amyl acetate process.	Catalyst Amberlyst - 15, temperature 373 K – 423 K.	Two design alternatives, coupled reactor/distillation column and reactive distillation column, were assessed. Total annual cost indicated that reactive distillation is 4 times more effectual than the coupledReactor/separator for the	[19]

		amyl acetate process.	
Dehydration of Glycerol.	metallic catalysts including alumina, magnesium, copper ruthenium, nickel, platinum, palladium, , raney nickel, and copper-chromite, temperature 513 K,pressure 98kPa.	In this study, dehydration of glycerol to acetol has beenVerified using different catalyst. High yield was obtained using copper-chromite catalyst in semi-batch reactive-distillation.	[20]
Methyl acetate production by using RD.	sulfonic acid ion exchange resin at a pressure of 1 atm.	Found an optimum value for the reflux ratio, producing a design with the minimum number of stages. And also verified the equilibrium design with a kinetic simulation.	[21]
Effect of the Chemical Equilibrium Constant on the Design of RD Columns.	Temperature 366 K.	Studied effect of the chemical equilibrium constant on the design of reactive distillationColumns. As equilibrium constant at 366 K decreases, higher numbers of reactive trays are required and operating pressures are lower. Total annual cost is higher.	[22]
Glycerol ethers synthesis from glycerol etherification with tert -butyl alcohol.	Amberlyst 15, temperature 298 K.	Concluded that Langmuir–Hinshelwood activity based mode found as best kinetic model also suitable configuration consists of 6 rectifying stages and 6 reaction stages without stripping stage.	[23]
Recovery of lactic acid.	Amberlyst-15, temperature 365-375 K.	Performed esterification, distillation and hydrolysis in a single unit. Lactic acid can be recovered by batch reactive distillation from its aqueous solution.Pseudo-homogeneous model was used for the determination of reaction kinetics.	[24]
Ethyl Acetate Production.	Temperature 373 K - 378 K.	Studied RD unit of capacity 60 TPD, column has 48 bubble-cap	[25]

		trays. Concluded that RD is not suitable only for new plant but also revamping of existing plant.	
Production of Butyl Acetate.	Temperature 366 K, pressure 15 psia.	Design and control of conventional and RD processes for the production of butyl acetate from methyl acetate have studied. RD tower has 30 trays. PI controller effectively control both process.	[26]

4 Reactive Absorption

The absorption of gases in liquid solution with chemical reaction is important industrial operation for production of basic chemicals and removal of harmful substances from gas streams. This process becomes important for purification of gases to high purity. As an industrial process, the most commonly encountered use of reactive absorption is for the separation and/or purification of a gas mixture by the absorption of part of the mixture (e.g., CO_2, H_2S, NOx and SOx) in a solvent that is regenerated afterwards. However, apart from gas cleaning,Reactive absorption is also applied in the production of bulk chemicals, such as nitric and sulfuric acid. More recently, reactive absorption using green catalysts offer great opportunities for manufacturing fatty esters, involved in specialty chemicals and biodiesel production.

Unlike physical absorption reactive absorption give high throughput for moderate pressure and it does not required large amount of solvent. The advantage of reactive absorption realized only region of low gas phase concentration. Limitation of reactive absorption is less efficiency due to heat liberated by chemical reaction and problem in solvent regeneration. Most of the reactive absorption operations are steady state. It is also complex rate controlling process that occurs far from thermodynamic equilibrium [27].

Review of the process is given in table 3

Table 3. A summary sheet of reactive absorption processes

Purpose	**Process conditions**	**Comments**	**Reference**
analyzing the effects of primary amines used	Temparture 1123K- 1273 K, Monoethanolami	Studied optimization of the process parameters, for reduce the energy consumption for the solvent regeneration.	[28]

monoethanolamine for CO_2 post combustion capture.	ne (MEA).	Two cases MEA 30 and 40 wt. % compared and process efficiency 90% obtained in both cases.	
Reactive Absorption of Ammonia and Carbon Dioxide.	Carbonated Ammonia Solution, temperature302-306 K.	Reactive absorption of carbon dioxide and ammonia in partially carbonated ammonia has investigated in pilot plant column. Result revealed that carbon dioxide absorption rate depend upon concentration of free ammonia.	[29]
reactive absorption for biodiesel.	Temperature 408 - 433 K,sulfated zirconia as acid catalyst.	Studied biodiesel process basedon FFA esterification in a reactive absorption column using solidacids as green catalysts. High purity and yield achieved. AspenTech Aspen Plus and ICARUS, were used development and evaluation of process.	[30]
reactive absorption for post combustion CO_2 capture from NGCC (natural gas combined cycle.	CO_2 concentrations in the range 1-30 volume %.	Studied CO_2 capture unit using reactive absorption using novel generic solvent.Simplest possible capture process configuration used. Improvement Capture process was reported.	[31]
Carbon Dioxide Capture by Reactive Absorption with Novel Solvents.	partial pressure of CO_2 35-135 mbar,CESAR1, CESAR2 solvent.	Investigated new solvents, CESAR1 and CESAR2, for capturing CO_2. Both solvent show 20 % reduction in regeneration energy and 45 % reduction in solvent flow rate.	[32]
Modelling of the reactive absorption of CO_2.	temperature313 K,MEA solvent.	Presented a rate-based model for the reactive absorption of CO_2 using amines. The comparison of the simulation results with the experimental measurements validates rate based model.	[33]
reactive Absorption Processes in Sour Gas Treatmentusing RigorousModelling.	temperature 298 K, KOH solvent	Developed model validated by vapour-liquid equilibrium data and process optimized such way that decrease of 30 % operating cost.	[34]

5 Reactive Membrane Separation

Membrane separation technology widely used in chemical, biochemical and environmental technology.This process saving energy and cost as compared to other technology such as reactive distillation, reactive absorption etc.Reactive membrane separation, a hybrid process combing chemical reaction and membrane separation.Membrane besides providing separation function also provide enhanced selectivity and yield. The process attains its wide feasible product spectrum by means of selective membranes, but little literatures provide perspectives on the feasibility analysis of reactive membrane separation, because of the even more complicated blend of chemical reaction and mass transfer kinetics. Ren[35] studied reactive membrane separations for power-generation applications. Also Stankiewicz [36] mention possible application areas of catalytic membrane reactors in the base-chemicals sector include: Dehydrogenations, e.g. ethane to ethene, ethylbenzene to styrene methanol to formaldehyde, Methane steam reforming, Water/gas shift reaction, Selective oxidations, e.g. propane to acroleine, butane to maleic anhydride, ethylene to ethylene oxide, Oxidative dehydrogenations of hydrocarbons, Oxidative coupling of methane, Methane oxidation to syngas. Review of reactive membrane is given in table 4.

Table 4. A summary sheet of reactive membrane processes

Purpose	**Comments**	**Reference**
enzymatic transformation.	Investigated confluence of two of Terry's thematic research streams by demonstrating that membrane vesicles produced by dissimilatory metal-reducing bacteria from the genus *Shewanella*catalyse the enzymeatic transformation and precipitation of heavy metals and radionuclides.	[37]
Test the ability to ferment sugars in membrane support- ed Liquid - liquid extracted and amm-onium hydroxide conditioning(AHC).	Studied detoxification of dilute-acid-pretreated corn stoverhydrolysate using reactive membrane extraction. Technique improves ethanol yields and rates .the process having disadvantages like high cost and complex process	[38]
water purification.	Investigated nanostructured, two- membrane stacked system, harmful organic contaminants can be degraded through the addition of a substrate, glucose, which is enzymatically converted to H_2O_2, thus eliminating the need for additional chemical reagents.	[39]
As(III) removal by hybrid reactive membrane.	Investigated the removal of arsenite (As (III)) was using a combined ozonation-reactive ceramic membrane incorporated with iron oxide	[40]

	nanoparticles (IONs).	
ethylene/ethane separation.	Investigated ethylene/ethane separation using Nafion - poly composite membranes. The membranes were verified with mixtures of ethylene and ethane at the total feed pressures ranging from 760 to 1900 mmHg.	[41]

6. Reactive adsorption

Chemical reaction and separation both occur at same time in reactive adsorption. The application off this technique now used in food, refining, environmental, biotechnology etc. Fixed or fluidized beds are used for this purpose. To overcome equilibrium, reactor size are compact, alternative to reaction distillation these are some advantageous of reactive adsorption and large quantity of adsorbent and catalyst required for this process is disadvantageous of this process. Research study related this summarized in Table 5

Table 5. A summary sheet of reactive adsorption processes

Purpose	**Process conditions**	**Comments**	**Reference**
Adsorption of Dibenzothiophene.	adsorbents, two carb- ons with and without sodium, at room temperature.	Investigated that sodium candispersed on the surface of a carbonaceous support in the process of DBT removal from hexane solution.As a result of mono and disodium thiophene metalates along with sodium sulphides are formed.	[42]
IR study of reactive adsorption of organic molecules over metal oxides.	different metal oxide surfaces.	The case study discussed above show that IR spectroscopy allows to give a contribution to the identification of the reaction mechanisms and surface reaction path ways in oxide-catalyzedoxidations.	[43]
Reactive adsorption of mustard gas.	zirconium (hydr)oxide/graphite oxide composites as adsorbent.	Investigated Zirconium oxide and its composites with various amounts of graphite oxide as reactive adsorbents of the mustard gas surrogate.	[44]
Reactive adsorption of acidic gases.	copper-containing HKUST-1) and	Studied the use of metal–organic framework and graphite oxide for	[45]

	graphite oxide as adsorbent.	removal of gases, hydrogen sulphide (H_2S) and nitrogen dioxide. This materials show high adsorption capacities in the presence of humidity.	
Reactive Adsorption of Ammonia on Graphite Oxides.	Adsorbent Graphite oxide.	Investigated performance of graphite oxides as the ammonia adsorbent caused by the different amounts of water present within the interlayer space. Water improves the amount of ammonia adsorbed.	[46]
Mercury Removal from Oil.	Coppersulfide on alumina or silicaalumina, silver, silver/gold, nickel or copper on support , sulfur on zeolites as adsorbents and temperature 366 - 477 K .	Investigated different adsorbent discussed in process condition was used to remove mercury from oil up to 99 %. This process can apply for refineries, LNG plant and petrochemical complex.	[47]

7. Reactive crystallization,

Reactive crystallization has been investigated by numerous research groups. It involve simultaneous reaction and crystallization. This process generally used in fine chemicals and pharmaceuticals. Processes of industrial relevance include liquid-phase oxidation of para-xylene to terephthalic acid, the acidic hydrolysis of sodium salicylate to salicylic acid, and the absorption of ammonia in aqueous sulfuric acid to form ammonium sulfate [48]. Reactive precipitation in high-gravity field has successfully been used for the production of nano-size cubic particles of $CaCO_3$. Ultra-fine particles with the mean size of 15- 40 nm and a very narrow size distribution were produced by carbonation of lime suspension in a Rotating Packed-Bed Reactor[49]. The reaction times in RPBR were 4- to 10-fold shorter than the corresponding reaction times in a conventional stirred-tank unit. Research related this summarized in table 6.

Table 6. A summary sheet of reactive crystallization processes

Purpose	**Process and conditions**	**Comments**	**Reference**
ReactiveCrystallization of L-Glutamic Acid (process control).	Semi-batch crystallization process and PID-controller used.	The obtained results showed that the feedback processcontrol approach developed for semi-batch precipitationAllows control of both the form of the polymorphs and the crystal size distribution. On the basis of the results obtained,the supersaturation level at the nucleation moment affects almost linearly the polymorphic composition	[50]
Reactive crystallization of nickel hydroxycarbonate.	fluidized bed reactor used,inlet Ni concentration 100 ppm (1.725mM), bed height at zero flow 200mm used.	Studied fluidized bed reactor with added silica seeds of nickel hydroxy-carbonate crystals onto a silica sand surface. Using concept of distribution of local supersaturation, control the supersaturation in the bed.	[51]
RC of calcium carbonate polymorphs.	glass crystallizer with a jacket was used, temperature 323 K.	Studied reaction between calcium hydroxide andSodium carbonate. And crystallization behaviorsof the polymorphs on the operational conditions and mechanism of crystallisation.	[52]
Reactive Crystallization of Nanoscale Six-Line Ferrihydrite.	Continuous crystallization,pH 3.6,temperature 358 K.	Studied of six-line ferrihydrite formation by continuous reactive crystal ization.Six-line ferrihydrite formed more Compact and larger aggregates of smaller primary crystals.	[53]
Polymorph control of calcium carbonate by	Semi-batch type crystallization,pH6.9	Studied use CO_2/NH_3 micro bubbles as new reaction fields	[54]

reactive crystallization.	–12.0,temperature 298 K,	in crystal nucleation progresses. the polymorph change in $CaCO_3$ occurred.	
ReactiveCrystallization of $SrSO_4$.	pH was adjusted 3 to 7, temperature 298 K.	Studied the influence of polyethylenimine (PEI) on the controlled double - jet crystalliz- ation process of $SrSO_4$. PEI modify the resulting crystalline properties	[55]
Production of LY288- 6721.	Pilot-Scale Continuous Pro- duction, temperature 323 K	Studied continuous reactive crystallization process, confirming control of physical properties of the isolated solids. The crystallization provided a high product yield and purity, along with control of physical properties.	[56]
Reactivecrystallization of calcium carbonate.	three-stage column crysta- llizer, temperature 288 K.	Investigated use of multistage column crystallizer and operating parameters effect on particle size distribution.economic viewpoint these crystallisers operated at low cost as compared mixed suspension mixed product removal crystallizers..	[57]

8.Conclusion

In conventional approach, process must include the separation of mixtures and recycling of the reactants so cost of process involve is high. The rates of reactions leading to desired products are often too low to establish economically attractive processes. Also the energy efficiency of endothermic and exothermal reactions performed industrially is often not satisfactory.The conversion of many reactions of interest is limited. So reactive separation is promising technology for the solution of above discussed problems.Reactive separation processes increasingly active investigation and development in industry. It leads to increased efficiency, greater process output and safety, reduced maintenance and operating cost. But the possible disadvantage for implementation of reactive separation must be considered. Combining the reaction and separation increase the complexity of modeling of process, control of the process. Development in process increase higher initial cost. This lead to additional efforts necessary to develop this field in future.

Abbreviations

LH-	Langmuir–Hinshelwood
LHHW-	Langmuir-Hinshelwood - Hougen - Watson
RAB-	reactive absorption
RAD-	reactive adsorption
RC-	reactive crystallization
RD -	reactive distillation
RE -	reactive extraction
RMS-	reactive membrane separation
RS -	reactive separation

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