

# Advance esterification reaction under microwaves

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**Abstract-** Currently, Microwaves are widely used in chemical Industry to accelerate chemical reaction. Some research result has shown that microwaves heating can significantly accelerate the reaction. The purpose of this experiment was to synthesize of dibutyl phthalic by using butyl alcohol, phthalic anhydride and catalyst as a sulfamic acid under the microwave oven. Kinetic study of above system was carried out it is found that reaction is very fast under microwave oven. Use of microwave energy to heat chemical reaction leads to not only reduce the chemical reaction from hour to minute also reducing side reactions and increases the yield and improve the reproducibility.

**Index Terms-** microwave (MW); Phthalic Anhydride (PAN); butyl alcohol (BuOH); ETHYL ALCOHOL (EtOH)

## I. INTRODUCTION

Sulfamic acid is strong mineral acid existing in crystalline form. It is cheap. It is used to clean equipments and vessels in food industry to remove deposits, scales and oxide films. It is also to sulfonate organic substances like alcohol, phenol etc. As catalyst it can replace sulfuric acid which is widely used for esterification. Use of sulfamic acid which is widely used for esterification. Use of sulfamic acid as a catalyst for esterification has many advantages over sulphuric acid and other catalyst. Aim of present work was to investigate the use of sulfamic acid as a catalyst for esterification. Catalytic action of acids in esterification reaction is closely connected with H<sup>+</sup> ion concentration. The stronger and more completely ionized acids possess high catalytic activity. General mechanism of esterification with the use of catalyst is given below.

When co-catalyst Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) is used along with H<sup>+</sup> ions as main catalyst, reaction mechanism occurs as given below. From the mechanism proposed we find that hydrogen peroxide is regenerated. Hence small amount of hydrogen peroxide gives more conversion which has been experimentally verified. Nowadays, the important development in the industry is that many traditional chemical processing techniques are reaching their optimum performance while consumers demand's stretch and governmental regulations tighten. The central starting point is the knowledge of chemical processes, which cause activation of the reaction or deterioration of the quality of the products. With this knowledge and by combining several techniques in an intelligent way it is possible to accelerate processes and to reduce the chemical uses. Microwave technology can be very useful for chemical processing, because products are heated directly instead of by convection and conduction. This means a reduction of the total processing time, no overheating and degradation of the product, and preservation of the product quality. The purpose of the study is to investigate the microwave superheating phenomena and their impact in homogeneous chemical reaction by using microwave heating. The specific mode of microwave heating is also used to improve chemical process by the selective and volumetric thermal effect of microwaves on the heterogeneous catalytic reactions.

### EFFECT OF STRUCTURE ON ESTERIFICATION

The rate at which different alcohols and acids are esterified as well as extent of reaction are dependent upon the structure of molecule and type of radical present. With acetic acid at 155°C, the primary alcohols are esterified more rapidly. Under the same conditions the secondary alcohols react much slower and have lower limits of esterification. The tertiary alcohols react much more slowly and yields are generally low 1 – 10% of conversion at equilibrium. Tests with isobutyl alcohol at chain (acetic, propionic, butyric) can be esterified readily. Esterification limits of acid increase with increasing the molecular weight of an acid. The introduction of branched chain to the acid decreases the esterification rate and two branches cause still retarding effect. Double bond also has its retarding influence.

With anhydrous ethyl alcohol and hydrochloric acid, the rate of esterification of straight chain fatty acid from propionic to stearic is substantially constant. Branching of chain causes retardation.

### COMPLETION OF ESTERIFICATION

Because of esterification of alcohol and organic acid involve reverse equilibrium these reactions do not go to complete, Conversion approaching 100% are desirable and can often be achieved by simple method of upsetting the equilibrium by removing the one of the product formed either ester or water. In general, the esters are divided into three broad classes depending upon volatility of esters.

1. Esters of high volatility – such as methyl formate, methyl acetate. Ethyl formate having lower boiling point than those corresponding alcohols and therefore, can easily be removed from the reaction mixture.
2. Esters of medium volatility – these are capable of removing water formed by distillation e.g. ethyl, propyl, butyl, amyl, formate, propyl, butyl acetate. In some cases ternary mixture of alcohol, ester and water is formed.
3. Esters of low volatility – In this case these have several types of esterification. In the case of esters of butyl and amyl alcohol, water is removed as binary mixture with alcohol. To produce esters of lower alcohol, it may be necessary to add hydrocarbon such as

benzene and toluene to increase the amount of distilled water with high boiling point alcohol e.g. Benzyl an accessory liquid is required to remove the water by distillation.

#### EQUILIBRIUM

The fact that esterification reaction involve an equilibrium was established in 1862 by M.P.E Berthelot in his study of ethylalcohol-acetic acid system.

#### CATALYSTS

In absence of catalyst all esterification reactions are very slow. In present of catalyst however such as H<sup>+</sup> ions in liquid media or dehydrating oxides in gas phase , the point of equilibrium may reach very rapidly .The usual esterifying of from 1 to 5 % of the reaction mixture undergoing esterification .

The choice of proper catalyst for an esterification reaction is dependent upon several factors .the most common catalyst used are strong mineral acids but other reagents such as tin salt , silica gel and cation exchange resin

### II.LITERATURE SURVEY

In present work esters of phthalic anhydride and n butyl alcohol were prepared using sulfamic acid as a catalyst. Benzyl alcohol was esterified with various carboxylic acids using the same catalyst. V.V.Mashkova et.al.(1) , investigated sulfamic acid as a catalyst for the esterification process. They found that sulfamic acid present to the extent of 5% by weight of phthalic anhydride had the greatest catalytic effect. Esters of phthalic, maleic and adipic acid were prepared with sulfamic acid as a catalyst. The synthesis was conducted for the period of 25-40 min. the catalyst was then filtered off the conversion of acid to ester amounted to 96-98%. Beside sulfamic acid, various other catalysts have been investigated for the esterification of phthalic anhydride with the aliphatic alcohols.

In 1974 Ando sumio et. Al. (2) used metal ion complex as a catalyst for the esterification anhydride with alcohol. The mixture of 184 gm. Phthalic anhydride and 325 gm. Octanol was heated with stirring and removal of water. 2.96 gm. of 2 mgo- 6sio<sub>2</sub> was added at 115 c and mixture heated for 1hrs 40 min. at temperature 195-200 c. to give 99% conversion. N dibutyl phthalate was prepared by mogica et. Al. (3), using h<sub>2</sub>so<sub>4</sub> as a catalyst the reaction temperature was 140 c. Kaledkowska et. al. (6) esterified phthalic anhydride with butyl alcohol in two stages. First stage was non catalytic and used 3.5:1, BuOH- phthalic anhydride at reflux and gave 0-BuOOB UO<sub>2</sub> CC<sub>6</sub> H<sub>4</sub> CO<sub>2</sub> H. As soon as H<sub>2</sub>O was observed in still, 12.5 weight % of wofatit KPS was added and H<sub>2</sub>O removed continuously to give 96.9 % of (BuOC) 2 C<sub>5</sub> H<sub>4</sub> after 8 hrs.

Jiaojaslin et. al. (7) worked on esterification of phthalic anhydride using rare earths as a catalyst. They found that the catalytic activity of light trivalent rare earth oxide was higher than that of heavy rare earth oxide in esterification of phthalic anhydride of alcohol. Among the light rare earth oxides Tm<sub>2</sub>O<sub>3</sub> and Ho<sub>2</sub>O<sub>3</sub> were less active but Er<sub>2</sub>O<sub>3</sub> and Yb<sub>2</sub>O<sub>3</sub> were rather active. The oxides of rare earth elements with more stable electron configuration such as La, Gd & Lu were relatively inactive. Takashoki kyoko et. al. (8), investigated hydrous zirconium oxide as a catalyst for esterification of carboxylic acid with alcohols.

Guo minguo et. al. (9), have given the method for preparation of liquid catalyst for the esterification . Liquid catalyst was prepared by chemical treatment of rare earth compound for esterification of acid with alcohol. Vigorously stirring the mixture of pulverized rare earth compound with 200-400 gm H<sub>2</sub>So<sub>4</sub> at 40-90 c for 2-5hrs gave a catalyst which was used for esterification 66 g acetic acid with isopentyl alcohol at 140c for 100 to 142 hrs to give 97.6% isopentyl acetate. Esters of acetic acid were prepared by Xu Vishung et. al.(10) , using perflurosulonic acid resin as a catalyst. Refluxing alcohol (EtOH, MeOH, PrOH) with AcoH mol ratio alc:AcoH =1.04:1 in presence of perelurosufonic acid resin for 1 hrs gave 33.9-99.1 % of the corresponding esters . Esters can be prepared directly in good yield under mild conditions from corresponding carboxylic acid and alcohol using appels salt (I) (4,5 , dichloro 1,2,3,dithizolium chloride) and 2,6, lutidine (II) Folmer James J et. al. (11), Thus RhcH<sub>2</sub> COOH was treated with EtOH, I&II in CH<sub>2</sub>C<sub>12</sub> for 2hrs at -78 c and then overnight at room temperature to give BnoEt. Benzyl esters were prepared by Maraves Dusan et. al. (12), using zeolite or synthesis mordenite as a catalyst. Benzyl acetate, Butyrate were prepared in 99.5 to 99.9 purity by refluxing equimolar amount of benzyl alcohol and respedtive acid in heptanes containing zeolite or synthesis mordenite. Mr. Joshi (13) worked on esterification of Benzyl alcohol with acetic acid, propionic acid & bytyric acid .Catalyst used was ion exchange resine amberlyst 15, along with co-catalyst hydergen peroxide .Reaction time was kept constant for 20 minutes .Percentage conversion was 35-70, depended on catalyst concentration and mole ratio of reactants. R Sengupta et .al .(14) studied kinetic of liquid phase esterification of Benzyl alcohol with Butyric acid using cation exchange resion Agrion c-100 as acatalyst .Effect of parameters such as catalyst concentration ,partical size , temperature etc .on fractional conversion of acid was studied.

D. N. Sharama et .al . (15) Studied the kinetics of esterification of ethanol with propionic using Dowx 50W-XB cation exchange resin in the H<sup>+</sup> Form. The influence of hydrogen ion concentration of the catalyst, temperature, mole ratio of reactants & particle size of catalyst on the conversion of ethanol was investigated.

### III.EXPERIMENT

SYSTEM: PHTHALIC ANHYDRIDE -BUTYL ALCOHOL  
SYSTEM: PHTHALIC ACHYDRIDE – n BUTYL AICOHOL

#### Experimental Method :

Phthalic Anhydride (PAN) was esterified with butyl alcohol (BuOH) using Sulfamic acid as a catalyst . Total 16 batches of above system were taken .

First two batches were taken at two different sulfamic acid concentrations viz, 3% & 6% of total weight of reaction time & conversion. Mole ratio of PAN & BuOH was kept constant at 1:2.5 in both batches.

**Apparatus :**

A three necked flat bottom and liter capacity Flask, was used as a reactor. It was kept on rotamantle provide with heater & magnetic stirrer. One neck was provided with thermometer well. Middle was stoppered .Third neck was connected with reflux condenser with the water separator.

**Materials :**

BuOH & PAN both were L.R. Grade, of above 99% Purity, & manufactured by Loba chemi Pvt . Ltd.  
Sulfamic acid – Commercial grade supplied by M/S P J Chemicals.

**Procedure :**

Into the flask was charged a 185 gm BuOH (2.5mols) & 148 gm PAN (1mol) . Heating & stirring was started to dissolved PAN in to BuOH . After dissolving the PAN into BuOH. 10 gm of sulfamic acid was added into the reactor. Zero time was taken as a time when reflux commenced.The course of reaction was followed by the quantity of the water collected in the separator. For 100% conversion of 1mol of pan, quantity of water collected should be 18 ml, However it was found that after getting a 15 ml of water, the rate of evolution of water decreased considerably. It was found that some water was with sulfamic acid. The reaction was considered as completed. The reaction product was cooled to room temperature. Sulfamic acid was then filtered off.

**Washing**

Reacting mass was taken into 1 Lit. Beaker, to this raw water was added. Mixture was then stirred at room temp. For 4-5 min .The heterogeneous mixture was allowed to settle in to two layers, in the separating funnel. Lighter aqueous layer was discarded. Heavier oil layer was subjected to rewashing as described above. Purpose of washing was to remove the traces of sulfamic acid and unreacted PAN.

**Steam distillation**

Washed product was then subjected to steam distillation to remove the unreacted BuOH. 10 gm of sodium carbonate was added to washed product before steam distillation. Steam distillation was carried out at atmospheric pressure. Steam was produced in pressure cooker (2 Lit. Volume). The course of steam distillation was followed by smelling the distillate. Steam distillation was stopped, when no smell of alcohol was found in distillate. Steam distilled product which contains condensed steam was allowed to separate into two phases. Both phases were separately received. Aqueous phase was discarded. The oil phase which is final product free from Sulfamic acid, PAN and BuOH, was weighed. Purity of final product was determined by measuring the Refractive index and SP. Gravity of final product. These properties for pure Dibutyl Phthalate, (1.BP) are

R.I was	1.491-1.492
Sp-Gr,	1.045-1.048

**Batch 3**

The catalyst used in batch 2, was reused in batch 3. The quantities of PAN & BuOH were same as that in Batch2. Experimental procedure was same as explained above.

**Batch No. 4,5,6,7**

Four batches were taken at different mol ratio of PAN & BuOH. Moles of PAN and concentration of catalyst was kept constant in each of the four runs. Following mole ratios of PAN & BuOH were taken.

Batch No.	Mol Ratio PAN:BuOH	% of Excess BuOH	Sulfamic Acid Concentration
4	1:2	0 %	3 %
5	1:2.2	10 %	3 %
6	1:3	50 %	3 %
7	1:4	100 %	3 %

Table 1. Experimental procedure was same as described for batch 1.

**Batch no. 8**

148 gm PAN & 185 gm BuOH mol ratio was taken for the esterification using H2So4 as the catalyst. Purpose was to compare the catalytic effect of sulfamic acid to that of H2So4.

### Batch no. 9

148 gm PAN was esterified with 148 gm of BuOH (PAN: BuOH= 1:2) using 8.9 gms of sulfamic acid. After completion of the reaction, the reaction mass was maintained at 200°C for 1 hr. After cooling to room temp., reaction product was washed & steam distilled as described for batch 1.

### Batch 10 and 11

1 mole of PAN was esterified with 2 moles of BuOH (PAN: BuOH=1:2) in 200 ml of xylene as the solvent. Experimental procedure was same as described for batch 1.

### Batch no. 12,13,14

Purpose was to determine how much alcohol (unreacted + excess) can be recovered. 148 gm (1 mol) of PAN and 185 gm (2.5 mols, 25% excess) BuOH was taken in each batch. The quantity of sulfamic acid was also kept const in each batch.

#### Alcohol recovery

Distillate from steam distillation contains two layers.

Lighter alcohol layer was separated from water layer and weighed. Salt was added in water layer and kept overnight to settle into layer and kept overnight to settle into two layers. Alcohol layer was removed, in this way dissolved alcohol was removed from the water.

### Batch no. 15 and 16

Co-catalyst H<sub>2</sub>O<sub>2</sub> was used along with sulfamic acid, a main catalyst. Both batches were charged with 148 gm BuOH (PAN: BuOH = 1:2.5) 10 gm sulfamic acid and 10 ml of 30% H<sub>2</sub>O<sub>2</sub> solution. Reaction time for one batch was 25 min & same for the other batch was 50 min. Both batches were carried out at total reflux. Procedure of washing and steam distillation was same as described for batch I

## RESULT & DISCUSSION

Ester obtained in each batch was almost pure.

R.I	1.491-1.492
Sp-Gr	1.045-1.048

The conversion of PAN to ester is given by ,

$$\% \text{ Conversion} = \left( \frac{\text{Weight of Ester Obtained}}{\text{Weight of ester for 100\% conversion}} \right) \times 100$$
$$= \left( \frac{\text{Weight of Ester Obtained}}{278} \right) \times 100$$

### Batch 1 and 2

The results of batch 1 and 2 are given in table III. Results show that the conversion is almost same in both batches. However reaction time has decreased by 1 hr 15 min when concentration of catalyst was doubled.

### Batch no. 12, 13, 14

Purpose of Batch No. 12, 13, 14 was to determine how much alcohol (unreacted + excess) can be recovered.

Unreacted alcohol is given by

Unreacted alcohol (A) = 2 % 74 (1-x) gms, where x is % conversion

If B is alcohol in excess of the stoichiometric requirement.

Then maximum alcohol available for recovered = A + B = 2 x 74 (1-x) B gms

If C is actual quantity of alcohol recovered then,

% of alcohol recovered =  $C/A+B = C/2x74(1-x)+B$

**A.SYSTEM: PHTHALIC ANHYDRIDE (PAN) – n BUTYL ALCOHOL (BuOH)**

Catalyst: Sulfamic acid

Pressure: Atmospheric

Variables: Temperature , Mole ratios , catalyst concentration

Run No	Mole ratios of reactant PAN : BuOH	Catalyst Meq/gm of reactant	Reaction temp – c	Reaction time	%Conversions
1	1:2.5	0.309	-	3hrs 15 mins	84%
2	1:2.5	0.619	-	2hrs	86%
3	1:2.5	0.619	-	3.75hrs	84%
4	1:2	0.619	130-180	1.5hrs	89.2%
5	1:2.202	0.309	125-180	2.5hrs	88.1%
6	1:3	0.309	122-130	3.75hrs	89.9%
7	1:4	0.309	113-115	4 hrs	81.1%
8	1:2.5	0.612	118-155	30 min	93.6%
9	1:2	0.309	110-190	1.5 hrs	89.2%
10	1:2	0.309	110-190	2 hrs	88.12%
11	1:2	0.309	110-190	1 hrs	90.28%
12	1:2.5	0.618	140-150	2 hrs	91.00%
13	1:2.5	0.618	145-155	2 hrs	92.00%
14	1:2.5	.618	140-165	2 hrs	92.00%
15	1:2.5	0.309	115-120	25 hrs	53.00 %
16	1:2.5	0.309	115-120	50 min	57.00%

TABLE III

Table IV gives of alcohol recovered in each batch.

**ALCOHOL RECOVERY**

System : PHTHALIC ANHYDRIDE – n BYTYL ALCOHOL

Catalyst : Sulfamic acid

Co-catalyst : -

Pressure : Atmospheric

Run No.	Mol Ratio PAN : BuOH	Sulfamic acid Concentration	Reaction Temp. C	Reaction Time	Conversion	% of Alcohol Recovered
12	1:2.5	6%	140-150	2hrs	91%	52%
13	1:2.5	6%	145-155	2hrs	92%	62%
14	1:2.4	6%	140-165	2hrs	92%	54%

TABLE IV

**DSCUSSION OF RESULTS**

Table III shows that with increase in concentration of the catalyst the time of reaction decreases. Generally it is found that the rate of acid catalyzed esterification is proportional to acid or hydrogen ion concentration. The result is in agreement with above statement. It can be seen from Table III that the reaction temperature has decreased with increase in % of excess alcohol. Increase in % of alcohol in proportional to the temperature, the reaction mixture decreases its boiling point, since rate of reaction is more for the batches containing higher % of excess alcohol. It is evident from Table III (batches 9 and 10 ) that using xylene as solvent there is no considerable change in reaction time and % conversion .Batch no.4 was carried out with using solvent (xylene) other experimental conditions were same as given for batch 9 and 10 (with xylene ). Conversion and reaction time for batch 4 was nearly same as that of for batch 9 and 10.

Results of Table IV shows that % of alcohol recovery are not appreciable. However, alcohol recovery can be improved by the fractional distillation of water of distillate (which contains dissolved alcohol) Co-catalyst  $H_2O_2$  was used in batch no. 15 and 16 % of conversion is very less in both batches. However, for other system studied, it is found that use of co-catalyst increases conversion while reducing the reaction time.

Result of batch 8 in which sulfuric acid was used as catalyst, shows that 93% conversion can be achieved within 30 min. Use of sulfuric acid as a catalyst has many disadvantages. On other hand sulfamic acid offer many advantages over sulfuric acid and other catalyst. Considering the advantages of sulfamic acid as catalyst, the time of reaction and operating conditions, manufacturing of dibutyl phthalate using sulfamic acid as a catalyst is economically attractive.

## B)SYSTEM : PHTHALIC ANHYDRIDE-ETHYL ALCOHOL(EtOH):

Phthalic anhydride was esterified with ethyl alcohol to form diethyl phthalate (DEP)Two runs of above system were taken. In first batch cation exchange resin was used as catalyst .Second batch was catalysed by sulfamic acid .Comparison between two batches was made on basis of % conversion and quality of product.

### Batch 1

#### Appartus

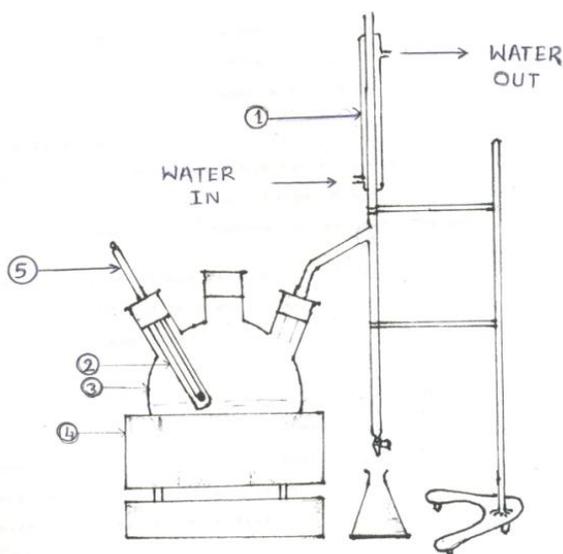
A three necked 1 lit capacity flat bottom flask was used as reactor. It was provided with thermometer well, stopper and reflux condenser. Reactor was placed on rotamentle provided with heater and magnetic stirrer.

#### Chemicals

Phthalic anhydride used in present investigation contained above 99% equivalent.Alcohol used contained 92% ethyl alcohol; remainder was water.Its sp. Gravity was 0.807. Cation exchange resin – Amberlite IR – 120 micporous cation exchange resin (-SO H). Exchange capacity 3.0Meq/gm

#### Procedure

Reactor was charged with 400 gm of PAN and 320 cc of ethyl alcohol. PAN was dissolved in ethyl alcohol. To the reactor 16 gm (4% by weight of PAN) of resin was added. Heating started.Zero time was taken as a time when reflux commenced. During the course of reaction after each 5 min 30 ml of alcohol was removed from the condenser and same quantily of fresh alcohol was added to the reactor. Initial temperature of the reaction was  $97^{\circ}C$ . It was raised to  $180^{\circ}C$  at the end of the reaction. Reaction was carried out for a hrs. Reaction was stopped by cooling the reaction mass to room temperature.Resion was filtered off from the reaction mass.



1-Condenser, 2-Thermometer well, 3-Reactor, 4-rotomantic,5- Thermometer

Figure 1: Experimental Set Up for System PAN-BuoH

#### Washing and steam distillation

Purpose was to remove unreacted PAN and ethyl alcohol from the reaction product was same as described for system PAN – BuOH. After washing and steam distillation, final product was weighed.

### Batch – 2

Catalyst used for batch 2 was sulfamic acid. Quantity of sulfamic acid used was 4 % (by weight) of PAN. Experimental procedure was same as described for batch 1.

### CALCULATIONS

% conversion is calculated as

$$\% \text{ Conversion} = \frac{\text{weight of final product}}{\text{weight of final product for 100\% conversion}} \times 100$$

$$= \frac{\text{weight of final product}}{222 \times \text{moles of PAN taken for reaction}} \times 100$$

RESULT: Result of batch 1 & 2 are given in Table V

SYSTEM : PHTHALIC ANHYDRIDE – ETHYL ALCOHOL

Pressure : Atmospheric

Temp : 97-180 C

Variable : Catalyst

Batch No.	Quantities PAN,EtoH (gm) (ml)	Catalyst	Quantity of catalyst (% of PAN)	Reaction time	% conversion
1.	400gms,320ml	Resion	4 %	8hrs	85.16%
2.	250gms,200ml	Sulfamic acid	4%	8hrs	94.90%

Table V

### DISCUSSIONS OF RESULTS

Final product of batch I was somewhat black in colour. This is because the temperature was raised above 128<sup>0</sup>C (a temperature at which resion decomposes). The temperature can be lowered by taking larger quantity of excess alcohol. This in turn increases the cost of separation. Product of batch 2 was clear liquid. This is because the maximum temperature reached (180<sup>0</sup>c) was well below the decomposition of sulfamic acid. The time of reaction and catalyst concentration was same in both batches. Higher % conversion in batch 2 indicates that sulfamic acid is more effective catalyst then ion exchange resion.

### BATCHWISE EXPERIMENTAL RESULT

BATCH	N-BUTYL ALCOHOL	PHTHALIC ANHYDRIDE	CATALYST
1	100% EXCESS (150 gms)	74 gms	SULPHAMIC ACID 1% by weight
2	200% EXCESS (222 gms)	148 gms	SULPHAMIC ACID
3	100 % EXCESS (150 gms)	148 gms	SULFURIC ACID

REACTION BATCH	HEATING USING CONVENTIONAL METHODS (MIN)	HEATING UNDER MICROWAVE OVEN (MIN)
BATCH 1	54	6
BATCH 2	45	5
BATCH 3	18	2

Comparison on the basis of time required for chemical reaction

**IV .KINETICS OF REACTION:**

Find out order of reaction

Sr No	Volume of sample (V1)ml	Burette reading (v2) ml	Concentration $N1 = \frac{N2V2}{V1}$	Time (min)
1	296	2	0.00675	2
2	296	4	0.0135	4
3	296	6	0.0202	6
4	296	8	0.0270	8
5	296	10	0.0337	10
6	296	12	0.0405	12
7	296	14	0.04729	14
8	296	16	0.0540	16
9	296	18	0.0608	18.30

From below graph we can evaluate another observation table for find order of reaction

Sr No	-rA	C <sub>A</sub>	Ln(-rA)	lnC <sub>A</sub>
1	2.5 x 10 <sup>-3</sup>	0.06	5.99	-2.81
2	4 x 10 <sup>-3</sup>	0.048	5.52	-3.03
3	2 x 10 <sup>-3</sup>	0.04	6.21	-3.21
4	4 x 10 <sup>-3</sup>	0.028	5.52	-3.51

From graph

$$\text{Slope} = \frac{y_2 - y_1}{x_2 - x_1}$$

$$N = \frac{6.8 - 6}{3.2 - 2.8}$$

$$= 2$$

Where N = order of reaction

Hence etherification is second order

Kinetics of reaction from graph

lnK = 2

Rate of reaction =k=7.38 min<sup>-1</sup>

$$-rA = kCA^n$$

$$-rA = 9.38(CA)^2 \text{ mol/lit min}^{-1}$$

**V.ADVANTAGES OF MICROWAVE OVEN:**

- High speed synthesis
- Reduce side reactions
- Increases yields
- Improve reproducibility
- Less electric power required
- Esters formed have no further Reaction mechanism

**VI. APPLICATIONS:**

For production of ester from animal fats for food industry

Excellent quality ester is formed used for biofuel  
It is used in paint industries

### VII CONCLUSION

- 1) The stirring process doesn't appear to be so notable under microwave irradiations as it is with conventional heating.
- 2) Rates of etherification reactions are influenced by types of catalyst heating temperature.

### VIII REFERENCES

1. V.V.Moslva N I Grishko N .I. Artamanova and T.B. Muravalyanskaya ' Kinetika I kataliz ' 12 , 1569 (1971)
2. Japan pat 70674338 (July 31 ,1970) Ando sumio and nalmato Takao
3. Rom pat .59087 (Apr .23 , 1969) Mongiuca Avram
4. Balozina L .M Nugatkina G.I and Kutsenko A .I ' Khim Prom st ' 580 1978 (8)
5. Nowokowski , lech and kaledkowska , mulgortala 'Chem.Engg. Tech . ' 58 , 48 , (1986)
6. Kaled Kowaska Malgorzatta , Nowaski lech and stepian Agust ' Chem .Engg . ' 53 , 101 (1991)
7. Jiao Zhaolin ,Pan Huifang and Luo Mingrun 'Chiuu xuebao ' 82 , 221 ,(1987)
8. Taka Shoki Kyoko ,shibagaki Makoto and Mitsushita Hagima 'Bull Chem . Soc Jap' 62 (7) , 2353 (1989)
9. Chin . Pat 10 58 520 (Feb 03 ,1993) Guo Mincua ,faming guandi and shenging Gongkai
10. Xu Yishuang Zhang Genmei , Ma Zhenxhong and zhao gengyum 'Huaxue Tonghao' 5 , 14 (1983)
11. Folmer James and Weinrab stevan ' Tetrahedron let . ' 34 (17) 2737 (1993)
12. Crech pat 258708 (jan 15 , 1989 ) Mravac Du san , Cihova Milina and Vojkto Jan
13. Project report of Ms . Joshi L.I.T , Nagpur
14. Project report of Mr. V.D.Gurudasani, LIT Nagpur
15. Bina H Shah , R Sengupta , S.S.Bhagade and G.D.Nageshwar ' Chemcon 89' page 182
16. O.M.Sharma , G.D.Nageshwar and P .S . Mene ' Ind Journal of Tech . ' 11, 360 (1973)
17. V.D.Gurudasani & M.S.Patil "Chemcon-10" Page No. 05

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