ISSN: 0975-833X

Available online at http://www.journalcra.com

International Journal of Current Research
Vol. 9, Issue, 07, pp.54511-54512, July, 2017

INTERNATIONAL JOURNAL OF CURRENT RESEARCH

RESEARCH ARTICLE

CONVERSION OF METHANOL AND DIMETHYL ETHER IN THE CONDITIONS OF ALKYLATION OF 2-METHYLPHENOL

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ARTICLE INFO

Article History:
Received 04th April, 2017
Received in revised form
29th May, 2017
Accepted 15th June, 2017
Published online 31st July, 2017

Key words:
2-Methylphenol,
Methanol,
Dimethyl Ether,
Alkylation,
Condensation,
Catalyst, PdCaY,
Recirculation.

ABSTRACT

The results of investigations of the side conversions of methanol in the conditions of alkylation of 2-methylphenol by methyl alcohol in the presence of the catalyst PdCaY are presented. It has been shown that the main product of the side conversion of alcohol – dimethyl ether is also able to develop the methylation reaction of *o*-cresol with average rate.

Citation: Muradov, M.M., Guseinova, I.G., Murshudlu, N.A., Agayev, A.A. 2017. "Conversion of methanol and Dimethyl ether in the conditions of alkylation of 2-methylphenol", *International Journal of Current Research* 9, (07), 54511-54512.

INTRODUCTION

The alkylation of phenols by methanol has received a practical application in the production of methyl homologues of phenol, in particular, cresols, xylenols and trimethylphenols (Dean Haymond, 2012; Kharlampovich, 1974). It is reported that (Zvi Rappopart, 2003) over solid catalysts along with alkyl,ation reaction there are a number of the side conversions of phenols and methanol, such as condensation, polycondensation of phenols with formaldehyde formed from methanol over acidic catalysts and also alcohol gasification at high temperatures and pressure. As a result, these undesirable conversions lead to decrease of yield and selectivity of process on purposeful products and to complication of technology and production.

In this report the results of investigations on methanol conversion in the presence of zeolite catalyst PdCaY, containing 1.0% mass of palladium are presented. This catalytic system shows the high catalytic properties in the alkylation reaction of 2-methylphenol by methanol.

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In the found reaction conditions (T-325^{0}C, ν-0,5h^{-1}, molar ratio of 2-methylphenol: methanol=1:1) a total yield of 2.6-, and 2.4- xylenols per reacted o-cresol is 88.5%, at conversion of 2-methylphenol for pass – 40.5%. A molar ratio of 2.6- and 2.4-isomers is 1:0,75. However, in these conditions, a total yield of these reaction products per reacted methanol doesn't exceed 60.0%, the rest part of the methanol is consumed for side reactions.

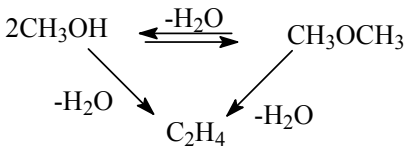
With the aim of study of the main side conversions of the methanol in the conditions of methylation of 2-methylphenol we have carried out the tests on alcohol conversion into PdCaY. The prepared results are presented in Table 1. It is seen that in the investigated conditions the methanol is subjected to the side conversions with formation of, mainly, dimethyl ether. In catalyzates there are also paraffins and olefins C_1- C_4, carbon oxide and dioxide, arenes and also water. In the presence of the studied zeolite one can isolate the primary stages of dehydration finishing by formation of ethylene

Table 1. Chemical composition of conversion products of the methanol on the catalyst PdCaY

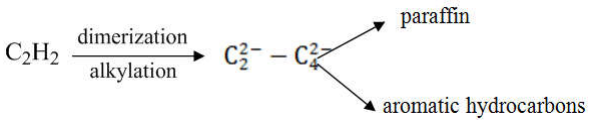
Name	Results			
Temperature, ^{0}C	325	325	350	350
Volume feeding rate of the methanol, h^{-1}	0,5	1,0	0,5	1,0
Prepared: mass %, including: methanol				
	51,9	57,0	28,0	49,6
dimethyl ether	33,0	29,7	47,6	33,8
water	12,5	11,6	18,6	13,2
CO_2	0,6	0,5	2,0	1,1
CO	0,4	0,3	1,0	0,8
C_2-C_3	0,7	-	1,2	0,5
losses	0,9	1,0	1,6	1,0
Methanol conversion, %	38,5	34,4	57,6	40,3
Yield of dimethyl ether per reacted methanol, %	95,0	96,0	88,9	93,1

Table 2. Results of interaction of 2-methylphenol by dimethyl ether on the catalyst PdCaY

Name	Results			
Temperature, ^{0}C	325	325	350	350
Volume rate, h^{-1}	0,4	0,4	0,4	0,4
Ether concentration in the initial mixture, mass %	16,0	23,0	16,0	23,0
Conversion of 2-methylphenol, %	14,0	19,5	18,5	23,5
Yield of reaction products per reacted 2-methylphenol				
2.6-dimethylphenol	61,2	55,0	48,0	44,5
2.4-dimethylphenol	10,2	12,3	14,0	15,5
2.4.6-trimethylphenol	11,8	10,5	18,0	15,0



and secondary stages of its further conversion on scheme



In addition, in the presence of this catalyzate there are the other methanol conversions about which the chemical composition of the prepared products evidences:

$CH_3OH \longrightarrow CO + 2H_2$

$CH_3OH + H_2 \longrightarrow CH_4 + H_2O$

$CH_2{=}CH_2 + H_2 \longrightarrow CH_3\text{-}\ CH_3$ и др.

In the process conditions on the zeolite PdCaY it mainly occurs methanol dehydration with formation of dimethyl ether. In the studied conditions a yield of ether per reacted alcohol is 88,9-96,0%. Consequently, on the zeolite PdCaY the secondary stages of the alcohol conversion are insignificant, about which the small quantities of forming hydrocarbons C_2 – C_3 evidence. In addition, it should be noted that in the conditions of alkylation of 2-methylphenol the above-mentioned alcohol conversions occur with less rate. Taking into account that the esterification reaction of the methanol can partially take place in the alkylation process of 2-methylphenol the alkylation of 2-methylphenol by dimethyl ether and also composition of the products forming in this case has been studied. The preparation of dimethyl ether was carried out by esterification of the methanol over catalyst PdCaY at 325^{0}C and volume feeding rate of the alcohol 0,5 and 1,0h^{-1} in the stationary reactor. The results presented in Table 2 show that with use of the dimethyl ether as methylating agent there are formed almost the same products as in the alkylation of 2-methylphenol by methanol. The exception is the absence of anisole and small quantities of forming 2.5-xylenol in prepared catalyzates. In addition, a degree of conversion of 2-methylphenol during alkylation by dimethyl ether is 14,0 - 23,5%, which almost in 2 times lower than in its alkylation by methanol. In this case a large yield of the side products is also observed. The presence of 2.5-xylenol and highly boiling products in the prepared catalyzates evidence about proceeding of the partial isomerization, condensation and it is possible a disproportionation in the absence of water vapor in the reaction mixture. Apparently, the water which is formed during alkylation of 2-methylphenol by methanol, adsorbing on the catalyst, decelerates a proceeding of these side reactions. It should be noted that the gaseous products prepared in interaction of 2-methylphenol with ether consist, mainly, of unreacted dimethyl ether. Thus, we have established the possibility of preparation of xylenols during alkylation of 2-methylphenol by dimethyl ether, especially, at low ether concentration in the initial mixture with sufficient selectivity. This allows to recycle the lesser forming gaseous products of the alkylation reaction of 2-methylphenol by methanol in the system, since the gas mixture consists, mainly, of dimethyl ether able also to develop the synthesis of dimethylphenols. In addition, this operation allows to exclude the gaseous emissions in the preparation process of the mixture of 2.6-, 2.4-xylenols on the basis of 2-methylphenol and methanol on the catalyst PdCaY.

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