International Journal of Chemical and Analytical Science
ISSN: 0976-1206
Research Article
www.ijcas.info

Novel Synthesis of 1,5-dibenzalacetone Using NaOH/ZrO_2-Montmorillonite as Cooperative Catalyst

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Synthesis of 4 analogue 1,5-dibenzalacetones through double cross aldol condensation has been conducted. Condensation was enganged by acetone with two different aldehyde at the same mole ratio at 10^0C for 3 hours with NaOH as catalyst. In the same time another reaction over NaOH / ZrO_2-montmorillonite catalyst was conducted. The purity and yield of both reaction were evaluated by thin layer chromatography technique. Yield of reaction result showed that cooperative properties of NaOH / ZrO_2-montmorillonite in cross aldol condensation was gained with more efficient impact compared to the use of only NaOH as homogeneous catalyst.

Keywords: cooperative catalyst, NaOH / ZrO_2-montmorillonite, 1,5-dibenzalacetones

INTRODUCTION

Aldol condensation reaction is usually performed to synthesize unsaturated αβ-carbonyl compounds with various advantageous such as chalcone, benzalacetone, etc. Synthesis of analogeous dibenzalacetone by reacting acetone and benzaldehyde and its derivatives is a well-known reaction [1,2]. Acetone which has Hα in both sides can be reacted in condensation with the same or different aldehyde. By the scheme, previous research investigated ultrasound-induced crossed aldol condensation of benzalacetone and several derivatives to obtain 1,5-dibenzalaseton [3]. Eventhough the reaction is an efficient reaction but it is time consumed due to two steps of reaction required. In simpler way, previous experiment was conducted by using acetone and two different aldehydes and derivatives but with low yield [4]. Refer to these works, in this paper, an efficient and novel method for producing high yield and purity of 1,5-dibenzalacetone is presented. Cross aldol condensation can be conducted under both acid and base condition and homogeneous form of catalyst [5]. Recently, heterogeneous catalyst has been attempted to replace the homogeneous catalyst refer to the more selective, stability and reusable characters [6,7. In this purpose for example, hydrotalcite was used as the most popular catalyst based on its anionic surface properties [8]. Started from this point of advantageous in using heterogeneous catalyst, many researches pay more attention to look for other solid catalyst instead of hydrotalcite.

Zirconium dioxide is a promising heterogeneous catalyst due to its thermal stability and reduction-oxidation properties. Potassium-modified ZrO_2 is heterogeneous base catalyst while in pillared form into montmorillonite has acid properties [9,10]. Due to surface acidity and its potential application as environmental friendly catalyst, it is interesting to take advantageous from immobilized ZrO_2 in montmorilonite. Refer to aldol condensation conducted by Ordomsky et al.(2010), an effort to use the active sites of immobilized ZrO_2 in montmorilonite can be achieved[6]. Bifunctional catalysis of Lewis acid and Broensted base of ZrO_2 / SiO_2 was reported to accelerate the reaction by abstracting Hα an aldehyde to form an enol structure for further reaction with C carbonyl from another acetaldehyde (Fig 1) [6].

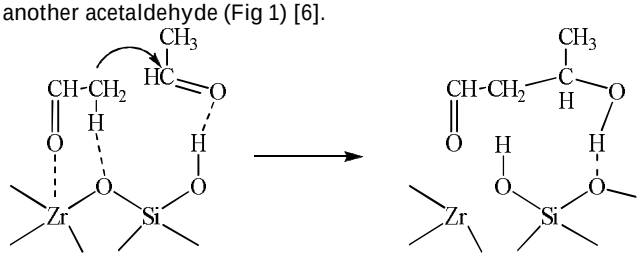

Fig 1. Aldol condesation of acetaldehyde over ZrO_2 / SiO_2 catalyst

MATRIAL AND METHOD

Materials used in this synthesis were acetone, benzaldehyde, 4-methoxybenzaldehyde, 3,4-dimethoxybenzaldehyde, 3-hydroxybenzaldehyde, HCl, methanol, ethanol, sodium hydroxide, ZrO_2-montmorillonite and aquadest. All chemicals were obtained from E.Merck in pure analytical grade while ZrO_2-montmorillonite was obtained from previous research [11]. Coloumn filler for chromatographic separation was prepared by silica gel 60 (230-400 mesh ASTM), TLC plate of silicagel 60 F_{254} and several solvents such as chloroform, hexane, ethyl acetate, and methanol. Analytical instruments were use for characterization purposes such as FTIR 8201 PC Shimadzu for FTIR-spectra investigation and (^{1}H and ^{13}C) Nuclear magnetic resonance (NMR) JEOL 500 with the frequency of 125 MHz together with HMQC and HMBC for structural analysis.

Synthesis of 4-methoxydibenzalacetone:. Two sets of reactions were enganged. The set number 1: consists of 0.6 g (0.015 mol) sodium hydroxide in 10 mL of ethanol-water (1:1), which was added by 0.1 g of ZrO_2-montmorillonite before the addition of 0.68 g (0.005 mol) 4-methoxybenzaldehyde, 0.53 g (0.005 mol) benzaldehyde and 0.29 g (0.005 mol) acetone

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Received 21-06-2012; **Accepted** 29-06-2012

sequentially followed by stirring at 10^0C (ice bath) for 4 hours in a flask with ethanol solvent. Reaction was kept for 18 hours in a refrigerator, continued by filtering the solution to obtain the solid precipitate which then dried. The set number 2 was the same reaction as number 1 but without addition of ZrO_2-montmorillonite. The product of both reactions was analyzed by using thin layer chromatography technique. The reaction using NaOH / ZrO_2-montmorillonite catalyst has resulted 1.32 g yellow precipitate with the target content of compound **1** is 47.48%, while the reaction without ZrO_2-montmorillonite resulted 0.24 g (7.29%).

The reaction that resulted highest value of yield was then separated by coloumn chromatography technique. Silica gel 60 (230-400 mesh) filled a 50cm in lenght x 2.5 cm in width of a coloumn was used with a mobile phase of ethyl acetate-hexane 1:9. Target compound was identified by thin layer chromatography (TLC) with the mobile phase of ethyl acetate-hexane 1:5 followed by elucidation of structure by using FTIR, ^{1}H NMR, ^{13}C NMR, HMQC and HMBC FTIR (KBr) (cm^{-1}): 3035 (aromatic CH), 2922 and 2842 (aliphatic CH), 1668 (C=O), 1423 and 1446 (aromatic C=C), 1175 and 1283 (C-O). ^{1}H NMR (500 MHz, $CDCl_3$, TMS) δ (ppm): 6.93 (2H, dd, *J* = 1.85 and 8.5 Hz, H3a and H5a), 6,97 (1H, d, *J* = 15.85 Hz, H2), 7.07 (1H, d, *J* = 15.85 Hz, H4), 7.4 (2H, dd, *J* = 1.85 and 8.5 Hz, H3b and H5b), 7.41 (1H, dd, *J* = 8.5 Hz, H4b), 7.58 (2H, dd, *J* = 1.85 and 8.5 Hz, H2a and H6a), 7.61 (2H, dd, *J* = 1.85 and 8.5 Hz, H2b and H5b), 7.72 (1H, d, *J* = 15.85 Hz, H5), 7.73 (1H, d, *J* = 15.85 Hz, H1), 3.8 (3H, s, H4a-OCH_3). ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm) : 56, 114.6, 123, 125, 127, 128, 129, 130.3, 130.5, 135, 143, 143.3, 161, 189.

Synthesis of 3,4-dimethoxydibenzalacetone: Two sets of reaction assigned as reaction number 1 and number 2. For number 1: a 0.6 g (0.015 mol) sodium hydroxide was solved in 10 mL ethanol-aquadest (1:1), followed by addition of 0.1 g ZrO_2-montmorillonite. 3,4-methoxybenzaldehyde of 0.83 g (0.005 mol), 0.53 g (0.005 mol) benzaldehyde and 0.29 g (0.005 mol) acetone were then added respectively under stirring at 10^oC (ice bath) for 4 hours. Similar procedure was done to synthesize 4-methoxydibenzalacetone, which then followed by ageing the result solution in refrigerator, filtering and drying. Reaction set number 2 was the same technique with reaction number 1 but without the addition of ZrO_2-montmorillonite. Characterization of the product was conducted by TLC with ethyl acetate–hexane 1:4 as mobile phase. A yellow precipitate of 1.99 g was obtained from reaction over NaOH / ZrO_2-montmorillonite and the target compound number **2** valued at 49.86% while from reaction without ZrO_2-montmorillonite, the product was 1.34 g (30.36%). Target compound was separated by coloumn cromatography technique in a coloumn with a 50 cm in lenght x 2.5 cm in width filled with stationary phase of 230-400 mesh silica and silica of 60 mesh for impregnation. As mobile phase, ethyl acetate-hexane 1:9 was employed. Characterization was also conducted by TLC technique using ethyl acetate-hexane 1:4. Structure Elucidation was performed by FTIR, ^{1}H NMR, ^{13}C NMR, HMQC and HMBC. FTIR (KBr) (cm^{-1}): 2933; 2837 (aliphatic CH), 1645 (C=O), 1514-1417 (aromatic C=C) and 1255-1139 (ether CO). ^{1}H NMR (500 MHz, $CDCl_3$, TMS) δ (ppm): 3.9 (6H, s, H3a-OCH_3), 6.81 (1H, t, *J* = 1.85 and 7.5 Hz, H3b), 6.83 (1H, t, *J* = 1.85 and 7.5 Hz, H5b), 6.89 (1H, t, *J* = 1.85 and 7.5 Hz, H5a), 6.94 (1H, d, *J* = 15 Hz, H2), 7.1 (1H, d, *J* = 15 Hz, H4), 7.14 (1H, d, *J* = 7.5 Hz, H2a), 7.2 (1H, dd, *J* = 1.85 dan 7.5 Hz, H6a), 7.33 (1H, d, *J* = 1.85 Hz, H6b), 7.41 (1H, dd, *J* = 1.85 and 7.5 Hz, H2b), 7.62 (1H, dd, *J* = 1.85 and 7.5 Hz, H4b), 7.69 (1H, d, *J* = 15 Hz, H1), 7.74 (1H, d, *J* = 15 Hz, H5). ^{13}C NMR (125 MHz, $CDCl_3$, TMS) δ (ppm) : 27.5, 56, 110, 111.2, 111.5, 123.3, 123.8, 125.5, 128.5, 129, 130.5, 143, 143.6, 149.3, 149.4, 151.6, 198.

Synthesis of 3,4,4'-trimethoxydibenzalacetone:. Two sets of reaction denoted as reaction number 1 and reaction number 2. Both reactions system consist of 15 mL ethanol-aquadest 1:1 with dissolved 0.6 g (0.015 mol) of sodium hydroxyde. At the reaction number 1, ZrO_2-montmorillonite was added before the addition of 0.83 g (0.005 mol) of 3,4-dimethoxybenzaldehyde, 0.68 g (0.005 mol) 4-methoxybenzaldehyde and 0.29 g (0.005 mol) of acetone respectively. The mixture was stirred for 3 hours at 10^0C in ice bath. The reaction number 2 was done at the same reaction number 1 without the addition of ZrO_2-montmorillonite. The mixture of both reactions were stored for 7 days in refrigerator and the precipitates obtained by filtering the resulted solution and drying the solid. TLC analysis of the products was enganged by using ethyl acetate-hexane (1:3). Reaction result of 2.45 g (47.69%) brownish-yellow precipitate compound **3** obtained over NaOH / ZrO_2-montmorillonite catayst, while the product obtained from reaction number 2 was 1.09 g (8.33%) in weight. Coloumn chromatography technique to separate the products was done using a 40 cm in lenght x 2.5 cm in width coloumn filled with silica 230-400 mesh and mobile phase of ethyl acetate-hexane 1:9. Characerization was conducted by TLC with the eluent of ethyl acetate-hexane 1:4.

The structure elucidation was performed by FTIR, ^{1}H NMR, ^{13}C NMR, HMQC and HMBC. FTIR (KBr) (cm^{-1}): 186.8 (CH alkene), 3070 (CH aromatic), 1674.2 (C=O), 1597 alkene 1512.19 (C=C aromatic), 1033-1273 (C-O), 1381 (CH_3). ^{1}H NMR (500 MHz, $CDCl_3$, TMS) δ (ppm): 3.8 (3H, s, H3a-OCH_3), 3.93 (3H, s, H4a-OCH_3), 3.94 (3H, s, H4b-OCH_3), 6.88 (1H, d, *J* = 8.55 Hz, H5a), 6.92 (2H, dd, *J* = 8.55 Hz, H3b dan H5b), 6.93 (1H, d, *J* = 15 Hz, H2), 6.98 (1H, d, *J* = 15 Hz, H4), 7.1 (1H, d, *J* = 1.85 Hz, H2a), 7.2 (1H, dd, *J* = 1.85 and 8.55 Hz, H6a), 7.57 (2H, dd, *J* = 1.85 and 8.55 Hz, H2b and H6b), 7.68 (1H, d, *J* = 15 Hz, H1), 7,7 (1H, d, *J* = 15 Hz, H5). ^{13}C NMR (125 MHz, $CDCl_3$, TMS) δ (ppm): 55.6, 56.1, 56.1, 76.9, 77.2, 77.4, 110, 123.2, 123.4, 124, 127.8, 128, 130.3, 142.9, 143.1, 149.4, 151.5, 161.5, 188.9.

Synthesis of 3-hydroxydibenzalacetone: Two sets of reaction denoted as reaction number 1 and reaction number 2. Both reactions system consist of 5 mL aquadest with dissolved 0.6 g (0.015 mol) of sodium hydroxyde. At reaction number 1, 0.1 g ZrO_2-montmorillonite was added to the solution under stiring to provide complete NaOH dissolved, while reaction number 2 performed without ZrO_2-montmorillonite. The solution were then added by 5 mL of ethanol, following by slowly addition of 0.005 mol of 3-hydroxybenzaldehyde, 0.005 mol of benzaldehyde and 0.005 mol of acetone, respectively. The reaction were then continued by stiring during 4 hours at temperature of 10^0 C in ice bath. The resulting precipitate was washed using H_2O, filtered and dried under bulb lamp. Composition analysis and characterization were done using TLC and TLC scanner (ethyl acetate-hexane 1:3). The reaction 1 resulted yellow precipitate compound **4** with reaction yield of 1.56 g (46.24%), while reaction 2 provided 0.83 g (13.35%). Separation of targeted compound from the side product was performed using coloumn chromatogrphy with a 40 cm in lenght x 2.5 cm in width coloumn filled with silica 230-400 mesh and mobile phase of ethyl acetate-hexane 1:9.

Identification was done by thin film chromatography with eluent of ethyl acetate-hexane 1:8. Structure elucidation was also done using FTIR, H-NMR, C-NMR, HMQC dan HMBC. FTIR (KBr) (cm^{-1}): 3444 (OH), 1668.83 (C=O), 1580 supported by 1493 (C=C aromatic), 1170-1249 (C-O). ^{1}H NMR (500 MHz, $CDCl_3$, TMS) δ (ppm): 8.6 (1H, s, H2a-OH), 6.93 (1H, dd, *J* = 1.75 dan 8 Hz, H4a), 7.2 (1H, d, *J* = 1.75 Hz, H4a), 7.23 (1H, d, *J* = 8 Hz, H6a), 7.24 (1H, d, *J* = 16 Hz, H2), 7.27 (1H, d, *J* = 8 Hz, H3a), 7.3 (1H, d, *J* = 16 Hz, H4), 7.4 (3H, t, *J* = 1,75 dan 8 Hz, H3b, H4b dan H5b), 7.73 (1H, d, *J* = 16 Hz, H1), 7.76 (2H, t, *J* = 1.75 dan 8 Hz, H2b dan H6b), 7.78 (1H, d, *J* = 16 Hz, H5. ^{13}C NMR (125 MHz, $CDCl_3$, TMS) δ (ppm) : 118.5; 115.6; 120.8; 126; 130.9; 126; 129.8; 143; 129.5; 143; 136; 137.5; 158; 189.

RESULT AND DISCUSSION

Characterization of synthesis results of compound 1, 2, 3 and 4 by using NMR spectrometer are listed in Table- 1- Table-4. Cross aldol condensation between acetone and two different aldehydes is presented in Fig.2.

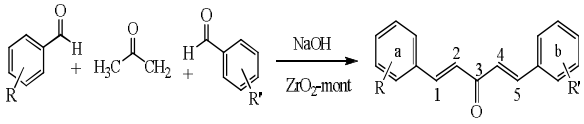


Fig.2. Cross aldol condenstion in synthesis of 1,5-dibenzalacetone.

Theoretically, synthesis of 1,5-dibenzalacetone which has different subtituent in ring a and b has low efficiency. This condition is caused by two step cross aldol condensation occured. The first cross aldol condensation left Hα that make it possible to undergo further cross aldol condensation. Therefore two kinds of aldehyde and some other side products were produced. Reaction efficiency was defined as high selectivity towards main product. As stated in introduction section, as an effort to increase efficiency heterogeneous catalysis was used in this research.

Table 1. ^{1}H, ^{13}C-NMR and HMBC data of compound 1($CDCl_3$)

No C	δH ppm	∑H, m, *J* (Hz)	δC ppm	HMBC (H→C)
1	7.74	1, d, 15.85	143.4	C1a, C3
2	6.97	1, d, 15.85	123	C2a, C6a, C3
3	-	-	189	-
4	7.08	1, d, 15.85	125	C1b, C3
5	7.71	1, d, 15.85	143	C3, C2b, C6b
1a	-	-	130.5	-
2a,6a	7.58	2, dd, 1.85 & 8.55	127	C1a, C1, C4a
3a,5a	6.93	2, dd, 1.85 & 8.55	114	C2a, C6a, C4a
4a	-	-	161	-
4a-OMe	3.8	3, s	56	C4a
1b	-	-	135	-
2b, 6b	7.61	2, dd, 15.85	130,3	C2b, C6b
3b, 5b	7.40	2, dd, 1.85 & 8.55	129	C1b, C3b, C5b
4b	7.41	1, dd, 1.85 & 8.55	128	C3b, C5b

Table 2. ^{1}H, ^{13}C-NMR and HMBC data of compound 2 ($CDCl_3$)

No C	δH ppm	∑H, m, *J* (Hz)	δC ppm	HMBC (H→C)
1	7.69	1, d, 15	143.7	C2a, C2, C3
2	6.94	1, d, 15	123.8	C3
3	-	-	188	-
4	7.1	1, d, 15	125,6	C1b
5	7.74	1, d, 15	143	C2b, C6b, C3
1a	-	-	151	-
2a	7.14	1, d, 1.85	110	C1, C1a
3a	-	-	149	-
3a-OMe	3.9	3, s	56	-
4a	-	-	149	-
4a-OMe	3.9	3, s	56	-
5a	6.89	1, d, 7.5	120	C1a, C4a
6a	7.2	1, dd, 1.85 & 7.5	123.4	C2a, C1, C1a
1b	-	-	135	-
2b	7.41	1, dd, 1.85 & 7.5	129	C1b, C6b
3b	6.81	1, t, 1.85 & 7.5	111,5	C4b, C1b
4b	7.62	1, dd, 1.85 & 7.5	128,5	-
5b	6.83	1, t, 1.85 & 7.5	110,6	C1b
6b	7.33	1, d, 1.85	129	C1b, C2b

Table. 3: ^{1}H, ^{13}C - NMR and HMBC data of compound 3 ($CDCl_3$)

No C	δH ppm	∑H, m, *J* (Hz)	δC ppm	HMBC (H→C)
1	7.68	1, d, 15	142.9	C3
2	6.93	1, d, 15	124	C1a, C3, C4
3	-	-	189	-
4	6.98	1, d, 15	123.4	C3
5	7.7	1, d, 15	143.1	C3, C6b
1a	-	-	128	-
2a	7.1	1, d, 1.85	110	C1, C4a
3a	-	-	149	-
3a-OCH_3	3.94	3, s	56	C3a
4a	-	-	151	-
4a-OCH_3	3.93	3, s	56	C4a
5a	6.88	1, d, 8.55	111.3	C3a
6a	7.2	1, dd, 1.85 & 8.55	123.3	C4a, C1
1b	-	-	127	-
2b, 6b	7.57	2, dd, 1.85 & 8.55	130.2	C5, C6b, C4b
3b, 5b	6.92	2, dd, 8.55	114.6	C1b, C4b, C5b
4b	-	-	161	-
4b-OCH_3	3.8	3, s	56	161

Aldol condensation reaction can be occurred in both acid and base environment. The use of some homogeneous catalyst such as chloride acid, sulfuric acid, sodium hydroxide and potasium hydroxide have been reported [4,12,13]. In other scheme, heterogeneous catalyst instead of homogeneous catalyst utilization has been evaluated as alternative. In this purpose, some material included hydrotalcite and potasium modified ZrO_2 have been reported as base heterogeneous solid catalyst [10,14].. As new idea, in this research the role of acid solid catalyst together with base catalyst has been tried to determine by using two catalysts of NaOH as base and ZrO_2-montmorillonite as acid solid catalyst. This idea based on theoretic approach that acid-base catalyst can be coordinative to enhance reaction efficiency [15]. Base catalyst of NaOH can be attack Hα of acetone to form a nucleophile. Lone pair electron in oxygen of carbonyl can be adsorbed by acid site of ZrO_2-montmorillonite catalyst so the carbon site will be more positive. This condition leads enolate acetone easier to attack carbonyl for condensation. Scheme of double

cross aldol condensation over NaOH / ZrO_2-montmorillonite is demonstrated in Fig-3 and Fig-4.

Table.4: ^1H, ^{13}C-NMR and HMBC data of compound 4 ($CDCl_3$)

No C	δH ppm	∑H, m, J (Hz)	δC ppm	HMBC (H→C)
1	7.73	1, d, 16	143.3	C3, C6a
2	7.24	1, d, 16	126.6	C1a
3	-	-	189	-
4	7.31	1, d, 16	126.6	C3, C1b
5	7.8	1, d, 16	143.4	C3, C2b, C6b
1a	-	-	137	-
2a	6.94	1, d, 1.75	118	C1a
3a	-	-	158	-
4a	7.2	1, d, 1.75	115	C6a
5a	7.27	1, d, 8	130.9	C1a, C3a
6a	7.23	1, d, 8	120.8	C4a
1b	-	-	136	-
2b, 6b	7.76	2, dd, 1.75 & 8	129.3	C2b, C6b
3b, 4b, 5b	7.4	3, t, 1.75 & 8	129.8	C3b, C4b, C5b

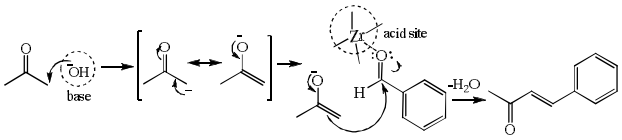


Fig. 3. First step of cross aldol condensation mechanism over NaOH / ZrO_2-montmorillonite.

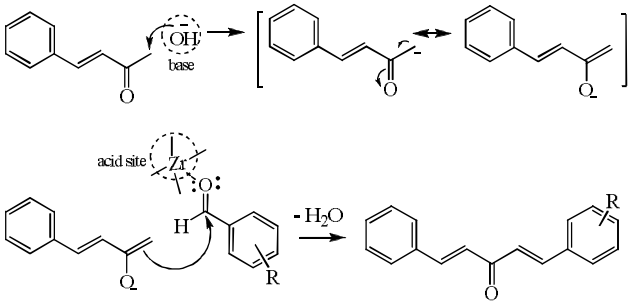


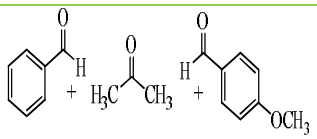
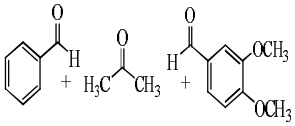
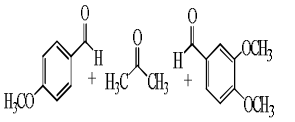
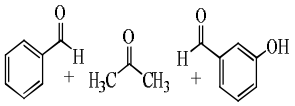
Fig. 4. Second step of cross aldol condensation mechanism over NaOH / ZrO_2-montmorillonite

Reaction mechanisms as depicted in Fig.2 and Fig.3 are potential to increase the formation of the product. Acid sites of ZrO_2-montmorillonite are easily to adsorb lone pair electron in oxygen of aldehyde carbonyl due to the lower steric hindrance effect. Therefore carbon in carbonyl has more positive profile compared to the carbon of ketone carbonyl and easier to be attacked by nucleophille. ZrO_2-montmorillonite that used in this reaction was the same material as reported in previous studies [11]. Important physico-chemical characters such as specific surface area, surface acidity and Zr content of material are responsible characters to accomodate base localization in order to activated the reaction orientation. The specific surface area of material is 96.75 m^2/g and the total surface acidity is 0.65 mmeq / g. With 3.34% wt. Zr content in ZrO_2-montmorillonite, Lewis acid sites from outer d-orbital of Zr provided bonding site of lone pair electron of oxygen from carbonyl as also showed by Broensted to Lewis acid ratio measurement at the value of 1.24.

Efficiency of the synthesis over cooperative NaOH / ZrO_2-montmorillonite was evaluated from the value of reaction yield based on following calculation:

$$yield = \frac{product\ weight\ x\ purity\ (area\ on\ TLC\ scanner)}{teorethical\ weight}\ x\ 100\%$$

Table. 5: Yield of synthesis result over NaOH and NaOH / ZrO_2-montmorillonite catalyst

No.	Starting materials	Catalyst	weight (g)/ purity (%)	Yield (%)
1		NaOH	0.24/40.12	7.29
		NaOH/ZrO_2-montmorillonite	1.45/43.22	47.48
2		NaOH	1.34/33.31	30.36
		NaOH/ZrO_2-montmorillonite	1.99/36.83	49.86
3		NaOH	1.09/12.38	8.33
		NaOH/ZrO_2-montmorillonite	2.45/31.54	47.69
4		NaOH	0.83/19.88	13.35
		NaOH/ZrO_2-montmorillonite	1.56/37.06	46.24

The result of the reaction yield was listed in Table 5, showed that the reaction using cooperative NaOH / ZrO_2-montmorillonite catalyst have resulted higher weight amount and purity of target molecules than the reaction that performed over only using NaOH. This is provided the fact that the use of cooperative catalyst leads to a more efficient and effectively cross aldol condensation reaction

CONCLUSION

Reaction of double cross aldol condensation has been conducted between acetone and two different aldehydes at the same mole ratio. The more efficient reaction mechanism was achieved by the combination of NaOH / ZrO_2-montmorillonite catalyst which attributed to the cooperative acid-base catalyst to minimize the formation of side product. The reaction yields of the reaction indicated that the surface acid sites and adsorption capability of ZrO_2-montmorillonite played the catalyst role in the double cross aldol condensation reaction with presence of NaOH in the reaction system.

ACKNOWLEDGEMENTS

Authors thank to Indonesian Science Institute (LIPI) Serpong, Indonesia for supporting NMR spectrometer Analysis.

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Source of support: Nil, Conflict of interest: None Declared