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MMZ_{NiY}-Catalyzed Tsuji–Trost Type of Reaction: A Selective Mono/Bis Allylation of Dicarbonyl Compounds

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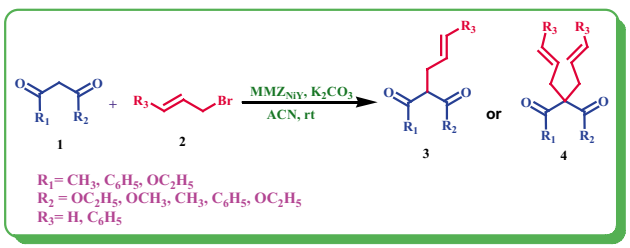
Abstract An alternative method to Pd-catalyzed Tsuji–Trost reaction is developed and it provides a simpler route for the selective synthesis of a broad range of *mono-/bis*-allylated and cinnamylated 1,3-dicarbonyl compounds using MMZ_{NiY} catalyst at room temperature. Product selectivity can be controlled by the proper choice of catalyst. The catalyst was also well characterized by SEM, TEM, HRTEM, EDAX and X-ray analysis. Other advantages of catalyst like its ease of preparation, functional tolerance and its reusability are also highlighted.

Graphical Abstract



Keywords Ni-catalyzed Tsuji–Trost reaction · 1,3-Dicarbonyl compounds · Allylation · Cinnamylation · $MMZ_{Ni\text{-}Y}$ zeolite

1 Introduction

Allylation of carbonyl compounds has been proved as a valuable tool for carbon chain elongation and thus extensively used for the functionalization of biologically active compounds which find pharmaceutical applications such as antibiotic, anticancer, DNA polymerase and HIV reverse transcriptase inhibitors [1–7]. Generally, Pd and its derivatives have been used to catalyze the allylic substitution at active methylenic compounds and these reactions are called as Tsuji–Trost reactions [8–18]. A few other metal complexes like Ru [19, 20], Ir [21, 22], Mo [23], W [24–26], Ce [27], Co [28, 29] and Bi [30] were also reported to catalyze the allylic substitution reactions. However, most of the described procedures suffer from the disadvantages such as the use of expensive metals, need of external ligands, tedious workup, longer reaction time, mixture of products and functional intolerance. As a consequence, development of new methods or materials to overcome these limitations is still in demand to make the procedure convenient and simple.

In recent years, the research activity for developing nanomaterials has grown exponentially owing to the fact that they offer better solutions to the challenges faced by various field. Niasari et al. have reported the preparation of various nanomaterials and investigated their catalytic properties [31–37]. Indeed, hierarchical nanoporous materials find many potential applications in the fields of drug delivery, hydrocarbon cracking, product selectivity [38–40] etc. In continuation of our interest in showing the multiple catalytic applications of hierarchically architectured MMZ_Y in organic transformations [41, 42], herein we report the selective allylation and cinnamylation of 1,3-dicarbonyl compounds.

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2 Results and Discussion

We have focused our initial studies on the optimization of reaction conditions and screening of suitable catalyst for the allylation of methylacetoacetate and the interesting results are summarized in Table 1. As there was no reaction in the absence of catalyst, different metal ion-exchanged zeolites were employed to find out the efficient catalyst for the present investigation. Bronsted acidic types of zeolites like HY and CaY produced a mixture of allylated products with very poor yields (Table 1, entries 2 and 7).

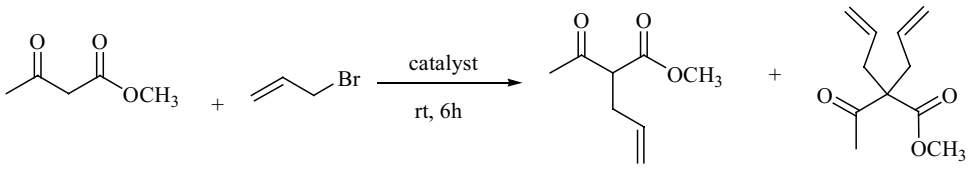
In the presence of alkali ion-exchanged zeolites also, we obtained a poor yield without significant selectivity (entries 3 and 8). However, in the presence of transition metal ions exchanged zeolite like Cu^{II}-Y, Ni^{II}-Y and Co^{II}-Y, the reaction proceeded faster and they piped out the allylated product from moderate to excellent yields (entries 4–6). Among them, Nickel-exchanged zeolite was found to be a very efficient catalyst which afforded 83% of *mono*-allylated product (entry 5). Our earlier reports on the use of MMZ_Y [41, 42] in other organic transformations have prompted us to prepare Ni^{II}-exchanged MMZ_Y and employ in the present study. Surprisingly Ni-exchanged MMZ_Y catalyst produced 96% of monoallylated product exclusively (entry 9). We have also examined the recycling of MMZ_{NiY} to see its efficiency which afforded the same yield even after two time usage.

The structural morphology of the hierarchical nanoporous MMZ_{NiY} zeolite was characterized by SEM, TEM, HRTEM, EDAX and powder-XRD methods (Figs. 1, 2). It clearly shows the uniform formation of Ni(II) species in the MMZ_Y framework of the catalyst with the particle size range of 0.5–1 µm. The XRD pattern of the MMZ_{NiY} are also found to be in good agreement with the parent zeolite framework structure. It clearly reveals that there is no change in crystallinity.

MMZ_{NiY} also shows its excellent tolerance toward the sterically crowded cinnamylation and the efficiency in selectivity toward *mono-*/*bis*-cinnamylated products (77–89%, Table 2).

The difference in reactivity of allyl/cinnamyl bromide with 1,3-dicarbonyl compounds is also attributed to the pka value of the substrates catalyzed by MMZ_{NiY}. In the case of keto-esters (Table 2, entries 1–8), *mono*-allylation is the major product (77–96%) due to their pka value are greater than 9, whereas in the case of diketones (entries 11–16), *bis*-allylation dominates (80–94%) due to their pka value are lesser than 9. However the exceptional case is dibenzoylmethane (entries 10 and 11), it results only a *mono*-allylation

Table 1 Screening of catalysts for the allylation of 1,3-dicarbonyl compounds



1a 2a 3a (mono) 4a (bis)

Entry	Catalyst	% Yield[a]	
		(**3a**) (mono)	(**4a**) (bis)
1	Neat	–	–
2	HY	19	14
3	NaY	21	18
4	CuY	61	29
5	NiY	83	11
6	CoY	70	22
7	CaY	24	19
8	CsY	19	06
9	MMZ_{NiY}	96	–

Reaction conditions: a mixture of methylacetoacetate (1 mmol), allyl bromide (2 mmol), catalyst (100 mg), K_2CO_3 (1 mmol) and acetonitrile (3 ml) were stirred at room temperature for 6 h

[a]Isolated yields

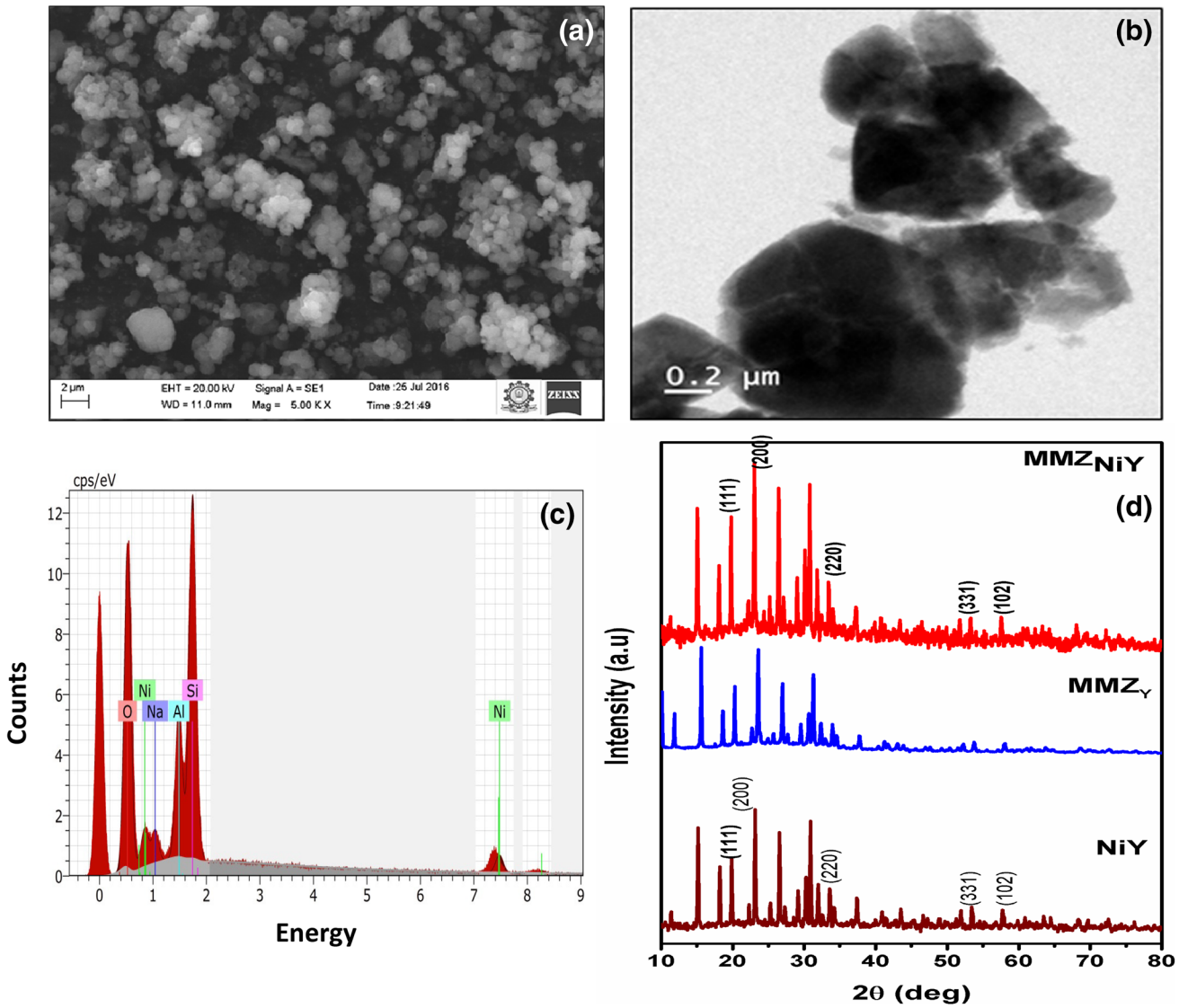


Fig. 1 **a** SEM. **b** TEM. **c** EDAX and **d** *p*-XRD spectrum of MMZ_{NiY} zeolite

which is a contrary observation with respect to other diketones employed in the present study. These interesting results are attributed to the cavity size of MMZ_{NiY}. It is worthy to note in the case of dibenzoylmethane where the nanocavities inside the MMZ_{NiY} arrests the free mobility of bulkier substrates and thus allows only *mono*-allylation/cinnamylation.

The role of MMZ_{NiY} in the allylation/cinnamylation can be easily understood through the proposed mechanism [18] that easy removal of acidic methylenic hydrogens of dicarbonyls by a simple base produces the nucleophilic methine carbon. At the same time, MMZ_{NiY} acts as a Lewis acid [39] and therefore it can readily coordinate with the double bond of allyl group and forms a Л-Ni-allyl complex. During the oxidative addition, the leaving group bromide is expelled and it gives η^2Л-allyl complex. Nucleophilic carbon then attacks the metal centre followed by a reductive elimination gives selectively allylated/cinnamylated product.

A careful study on the comparison of data with other reported catalysts has been made and the parameters are presented in Table 3. It clearly reveals that our catalytic system shows excellent selectivity with higher yields and moreover it is reusable when compared to other catalysts which requires the addition of pyridine, aryl phosphate ligand and argon atmosphere, reflux conditions, longer reaction time etc. It also includes other side reactions like dehydration, cyclization, mixture of products and failure to achieve cinnamylation.

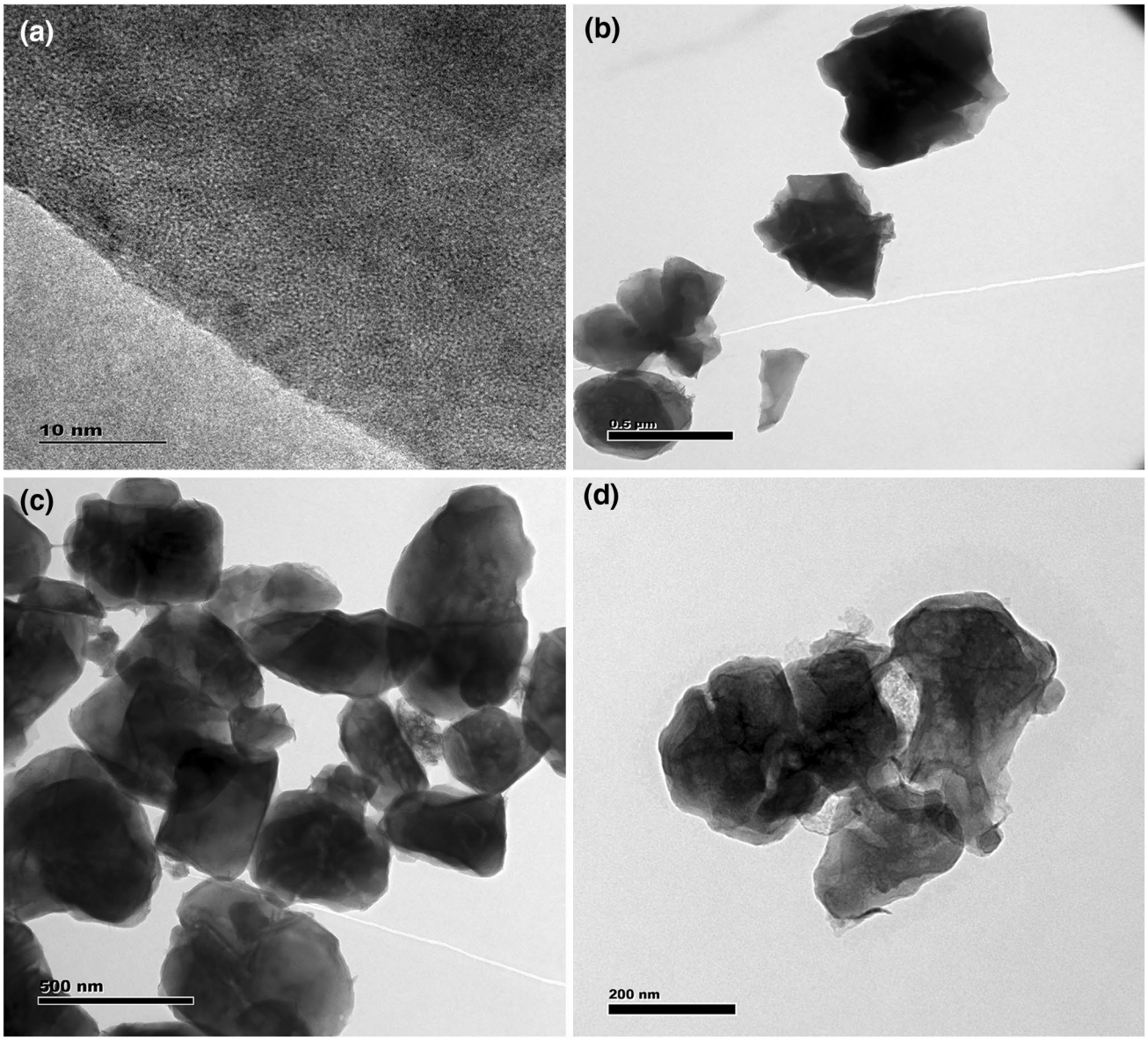


Fig. 2 HRTEM images of MMZ_{NiY} shows the presence of Ni ions within the cages and on the external surface of zeolite framework

3 Conclusions

In conclusion, we have developed a simple procedure for the selective *mono*/*bis* allylation and cinnamylation of various dicarbonyl compounds promoted by an inexpensive, reusable, non-toxic and environmentally benign novel solid acid MMZ_{NiY} catalyst. These reactions encompass a broad range of 1,3-dicarbonyls as well as simple and bulkier allylating agents. The other advantages of this procedure are no need of external ligands, use of a simple base, shorter reaction time and excellent selectivity with higher yields.

4 Experimental Methods

4.1 Synthesis of MMZ_{NiY} using Nanoporous Zeolite Y (MMZ_Y)

The nanoporous MMZ_Y zeolite was synthesized from the commercially available NaY zeolite according to the reported procedure [43, 44]. The as prepared MMZ_Y was stirred with nickel nitrate (100 ml, 10%) solution at 70 °C for 12 h. The exchange was repeated at least three times. Each time after exchange, the zeolite powder was washed

Table 2 Allylation and cinnamylation of various 1,3-dicarbonyl compounds using MMZ_{NiY} zeolite

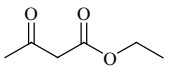
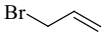
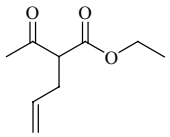
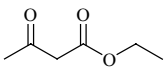
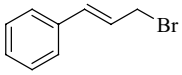
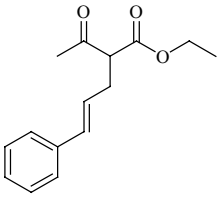
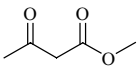
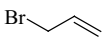
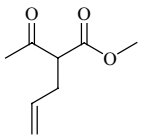
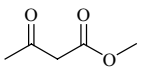
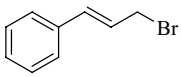
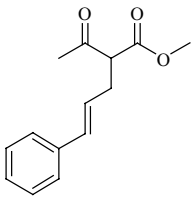
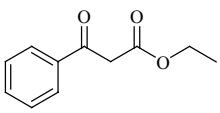
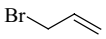
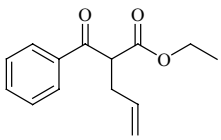
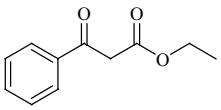
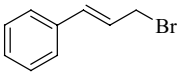
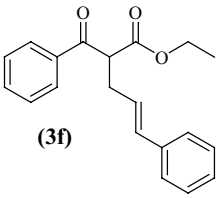
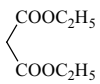
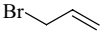
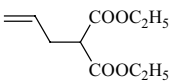
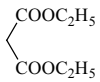
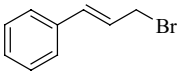
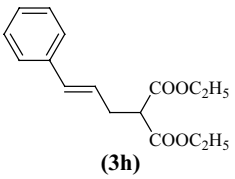
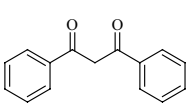
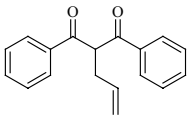
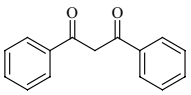
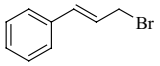
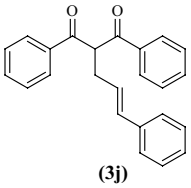
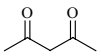
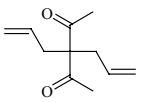
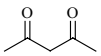
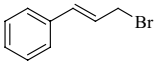
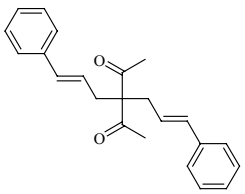
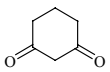
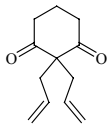
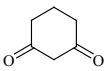
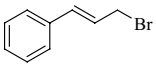
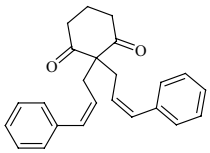
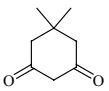
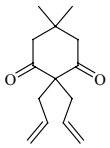
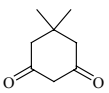
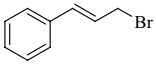
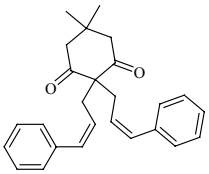
S.No	Substrate (1)	Allylating agent (2)	Major Product	Yield[a] (%) Mono (3)	Bis (4)
1.	 **(1a)**		 **(3a)**	92	--
2.	 **(1a)**		 **(3b)**	90	--
3.	 **(1b)**		 **(3c)**	96	--
4.	 **(1b)**		 **(3d)**	87	--
5.	 **(1c)**		 **(3e)**	89	--
6.	 **(1c)**		 **(3f)**	81	--
7.	 **(1d)**		 **(3g)**	93	--
8.	 **(1d)**		 **(3h)**	89	--

Table 2 (continued)

S.No	Substrate (1)	Allylating agent (2)	Major Product	Yield[a] (%) Mono (3)	Bis (4)
9.	 **(1e)**		 **(3i)**	85	--
10.	 **(1e)**		 **(3j)**	77	-
11.	 **(1f)**		 **(4k)**	--	92
12.	 **(1f)**		 **(4l)**	--	87
13.	 **(1g)**		 **(4m)**	--	94
14.	 **(1g)**		 **(4n)**	--	86
15.	 **(1h)**		 **(4o)**	--	92
16.	 **(1h)**		 **(4p)**	--	80

Reaction conditions: a mixture of 1,3-dicarbonyl (1 mmol), allyl bromide (2 mmol), K_2CO_3 (1 mmol), $MMZ_{Ni\text{-}Y}$ (100 mg) and solvent (3 ml) were stirred at room temperature for 6 h

[a]Isolated yield

Table 3 A Comparative study with other reported catalytic systems for the allylation of 1,3-dicarbonyl compounds

S.No.	Catalyst	References	Solvent	Temp.	Time (h)	Yield (%)
1	Pd(π-allyl)/DPCB	[9]	Pyridine	50 °C	3–12	>85
2	$Pd\{P(OC_6H_5)_3\}_4$	[11]	Toluene (Ar atm.)	80 °C	2–25	>80
3	$PdCl(\eta^3\text{-}C_3H_5)$/tppts	[14]	H_2O/AcOEt	rt	13–21	>70
4	$PdCl_2$, TBAB	[15]	THF/H_2O	Reflux	6–12	80–90
5	$Ru(Cp)(MeCN)_3[PF_6]$	[20]	CH_3CN	40 °C	16 h–2 days	>80
6	CTAN	[27]	CH_3CN, Et_3N	rt	4	60–80
7	$CoCl_2$	[29]	DCE	70 °C	8–12	30–67
8	$Bi(OTf)_3$	[30]	CH_3NO_2	100 °C	6	58–96

repeatedly with distilled water and then dried and activated at 450 °C before use.

Transmission Electron Microscopy (TEM) image of MMZ_{NiY} was obtained using a JEOL JEM-2100 LaB6 instrument equipped with the high-resolution (HRP), style objective-lens pole piece at an acceleration voltage of 200 kV. Scanning Electron Microscopy (SEM) images were taken by using FEI Quanta FEG 200-High Resolution Scanning Electron Microscope. HR-TEM images were recorded in JEOL—JEM-2100, at operating voltage of 200 kV with Lanthanumhexaborate filament using fermionic gun. The X–ray diffraction (XRD) patterns were taken with a Bruker D8 advance, Cu Kα radiation (40 kV, 30 mA). EDAX analysis was carriedout using Bruker instrument.

4.2 General Procedure for the Allylation/Cinnamylation of 1,3-Dicarbonyl Compounds Using MMZ_{NiY} Zeolite

A mixture of 1,3-dicarbonyls (1 mmol), allyl/cinnamyl bromide (2 mmol) and K_2CO_3 (1 mmol) were added to the reaction tube, which contains acetonitrile (3 ml) and 100 mg of freshly activated MMZ_{NiY} zeolite. The reaction mixture was stirred for 6 h. Products were extracted from the reaction mixture after the addition of 15 ml of dichloromethane solvent and allowed for 2 h stirring again. The catalyst was separated from the reaction mixture by simple filtration. Then, the solvents were removed by evaporation in a rotary evaporator. Proceedings of the reaction was constantly monitored by TLC [silica gel, pet ether:ethyl acetate (8:2)]. The products were purified by column Chromatography [using pet ether (97%):ethyl acetate (3%)].

All the isolated *mono*- and *bis*-allylated/cinnamylated carbonyl compounds were characterized by ^1H NMR and ^{13}C NMR spectra, were obtained on Bruker NMR (300 MHz) instrument in $CDCl_3$. GC-MS spectrum was recorded on Agilent-7820A GC system with 5977E MSD (DB-5 Column).

4.3 Spectral Data of the Isolated Products

4.3.1 Ethyl-2-acetyl-pent-4-enoate (3a)

Colorless Oil, ^1H NMR (300 MHz, $CDCl_3$): δ (5.72–5.63, m, 1H), (5.06–4.96, dd, 2H), (4.16–4.09, q, 2H), (3.46, t, 1H), (2.52, t, 2H), (2.17, s, 3H), (1.24, t, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 202.30, 169.0, 133.95, 117.20, 61.19, 58.96, 31.91, 28.87, 13.84.

4.3.2 Ethyl-2-acetyl-5-phenyl pent-4-enoate (3b)

Colorless Oil, ^1H NMR (300 MHz, $CDCl_3$): δ (7.31–7.29, m, 4H), (7.22, m, 1H), (6.45, d, 1H), (6.17–6.03, m, 1H), (4.21, q, 2H), (3.59, t, 1H), (2.77, m, 2H), (2.26, s, 3H),(1.25, t, 3H), ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.95, 136.83, 133.89, 132.51, 128.25, 127.13, 125.97, 125.92, 125.52, 123.61, 61.18, 59.40, 35.63, 31.26, 28.86, 13.85.

*4.3.3 Methyl-2-acetyl pent-4-enoate (**3c**)*

Colorless Oil, H NMR (300 MHz, $CDCl_3$): δ (5.81–5.67, m, 1H) (5.12, d, 1H), (5.03, d, 1H), (3.74, s, 3H), (3.55, t, 1H), (2.61, t, 2H), (2.24, s, 3H).

*4.3.4 Methyl-2-acetyl-5-phenyl pent-4-enoate (**3d**)*

Yellow Oil, ^1H NMR (300 MHz, $CDCl_3$): δ (7.35–7.3, m, 4H), (7.22–7.25, m, 1H), (6.47, d, 1H), (5.96–6.17, m, 1H), (3.75, s, 3H), (3.49, t, 1H), (2.71–2.46, m, 2H), (2.09, s, 3H).

*4.3.5 Ethyl-2-benzoyl-pent-4-enoate (**3e**)*

Brown Oil, ^1H NMR (300 MHz, $CDCl_3$): δ (8.01, d, 2H), (7.61, t, 1H), (7.48, t, 2H), (5.86–5.77, m, 1H), (5.15–5.02, dd, 2H), (4.4, t, 1H), (4.14, q, 2H), (2.78–2.72, m, 2H), (1.17, t, 3H), ^{13}C NMR(75 MHz, $CDCl_3$): δ 0.194.50, 169.37, 136.15, 134.47, 133.52, 128.71, 128.60, 117.40, 61.44, 53.91, 32.97, 13.99. GC-MS: Calcd. for $C_{14}H_{16}O_3$: 232.10. Found 232.1.

*4.3.6 Ethyl-2-benzoyl-5-phenyl-pent-4-enoate (**3f**)*

Yellow Oil, ^{1}H NMR (300 MHz, $CDCl_3$): δ (8.03, d, 2H), (7.61–7.49, m, 3H), (7.31–7.27, m, 4H), (7.22–7.21, m, 1H), (6.49, d, 1H), (6.26–6.16, dt, 1H), (4.47, t, 1H) (4.16, q, 2H), (2.93, m, 2H), (1.18, s, 3H).

*4.3.7 Diethyl-2-allylmalonate (**3g**)*

Yellow Oil, ^{1}H NMR (300 MHz, $CDCl_3$): δ (5.8–5.71, m, 1H), (5.03, d, 1H), (4.97, d, 1H), (4. 12, q, 4H), (3.19, t, 1H), (2.7, d, 2H), (1.3, t, 6H).

*4.3.8 Diethyl-2-cinnamyl malonate (**3h**)*

Colorless Oil, ^{1}H NMR (300 MHz, $CDCl_3$): δ (7.32–7.25, m, 4H), (7.22 7.20, m, 1H), (6.48, d, 1H), (6.18–6.14, m, 1H), (4.23–4.17, m, 4H), (3.49, t, 1H), (2.87–2.78, m, 2H), (1.25, t, 6H).

*4.3.9 2-Allyl-1,3-diphenyl propane-1,3-dione (**3i**)*

White Solid, mp 94–97 °C, ^{1}H NMR (300 MHz, $CDCl_3$): δ (7.95, d, 4H), (7.56, t, 2H), (7.43, t, 4H), (5.91–5.83, m, 1H), (5.30, t, 1H), (5.10, d, 1H), (5.02, d, 1H), (2.9, t, 2H).

*4.3.10 2-Cinnamyl-1,3-diphenyl propane-1,3-dione (**3j**)*

Pale yellow solid, mp 118–120 °C, ^{1}H NMR (300 MHz, $CDCl_3$): δ (7.97, d, 4H), (7.56, t, 2H), (7.44, t, 4H), (7.24, d, 4H), (7.18, m, 1H), (6.46, d, 1H), (6.27–6.21, m, 1H), (5.35, t, 1H), (3.02, t, 2H).

*4.3.11 3,3-Diallyl pentane-2,4-dione (**4k**)*

Yellow Oil, ^{1}H NMR (300 MHz, $CDCl_3$): δ (5.56–5.45, m, 2H), (5.15–5.09, dd, 4H), (2.65, d, 4H), (2.11, s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 205.47, 131.68, 119.04, 70.05, 34.69, 26.94.

*4.3.12 3,3-Dicinnamylpentane-2,4-dione (**4l**)*

Brownish Oil, ^{1}H NMR (300 MHz, $CDCl_3$): δ (5.56–5.45, m, 2H), (5.15–5.09, dd, 4H), (2.65, d, 4H), (2.11, s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 205.47, 131.68, 119.04, 70.05, 34.69, 26.94.

*4.3.13 2,2-Diallyl-cyclohexane-1,3-dione (**4m**)*

Colorless Oil, ^{1}H NMR (300 MHz, $CDCl_3$): δ (5.62–5.49, m, 2H), (5.06–5.01, dd, 4H), (2.54, q, 8H), (1.92, m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 210.27, 132.31, 119.22, 68.15, 40.77, 39.84, 16.31.

*4.3.14 2,2-Dicinnamyl cyclohexane-1,3-dione (**4n**)*

Yellow Solid, mp 134–136 °C, ^{1}H NMR (300 MHz, $CDCl_3$): δ (7.28–7.25, m, 8H), (7.23–7.19, m, 2H), (6.38, d, 2H), (5.99–5.93, m, 2H), (2.71, d, 4H), (2.54, t, 4H), (1.86, m, 2H).

*4.3.15 2,2-Diallyl-5,5-dimethyl cyclohexane-1,3-dione (**4o**)*

Colorless Oil, ^{1}H NMR (300 MHz, $CDCl_3$): δ (5.65–5.5, m, 2H), (5.1–5.03, dd, 4H), (2.59, s, 4H), (2.51, d, 4H), (0.96, s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 208.45, 132.33, 119.29, 67.80, 51.88, 38.66, 30.59, 29.52.

*4.3.16 2,2-Dicinnamyl-5,5-dimethylcyclohexane-1,3-dione (**4p**)*

Pale Yellow Solid, mp 149–150 °C, ^{1}H NMR (300 MHz, $CDCl_3$): δ (7.30–7.25, m, 8H), (7.21–7.18, m, 2H), (6.42, d, 2H), (6.03–5.97, m, 2H), (2.69, d, 4H), (2.57, s, 4H), (0.94, s, 6H).

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