CHAPTER 5

Synthesis of glycerol carbonate using bimetallic NiMgOx catalyst

CHAPTER 5

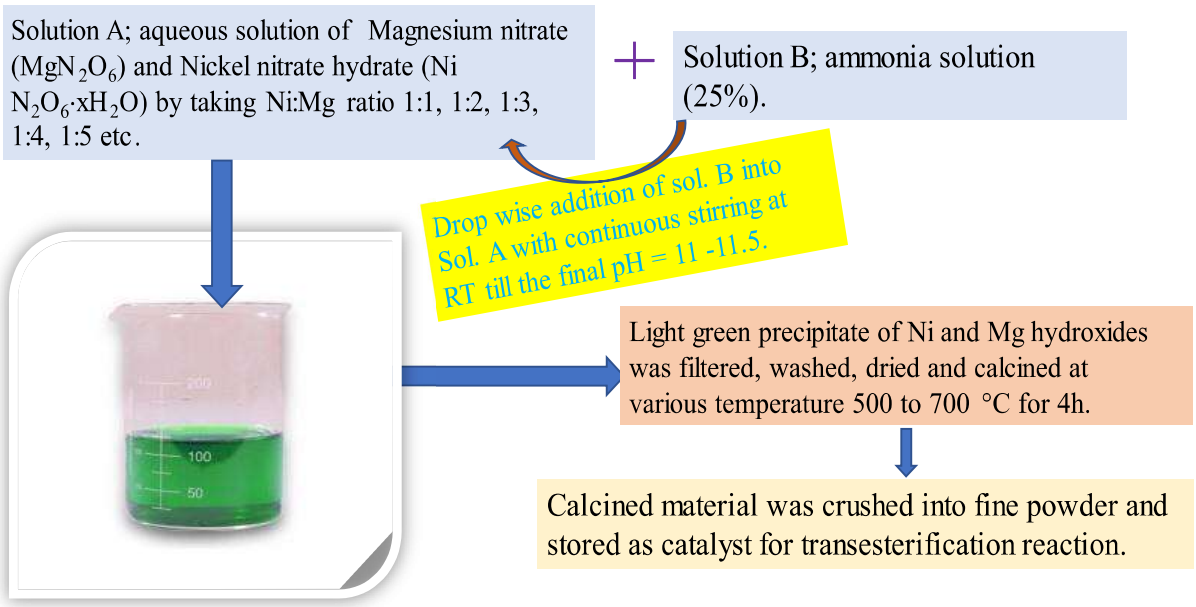
5.1 Introduction

Present chapter contains details of design of $NiMgO_x$ catalyst via co-precipitation route and its application in glycerol carbonate synthesis. So far there are no reports on application of $NiMgO_x$ in the valorization of glycerol and production of glycerol carbonate, the current study explores the potency of $NiMgO_x$ as an ideal catalyst for glycerol transesterification to glycerol carbonate. The influence of different molar ratios of Ni/Mg on the structural characteristics of $NiMgO_x$ and catalytic proficiency during conversion of bio-sourced glycerol assisted with dimethyl carbonate was studied through XRD, FT-IR, TGA-DSC, BET, FE-SEM EDX, XPS and batch catalytic tests. The consequence of several reaction parameters i.e., temperature, reaction time, catalyst loading, glycerol to DMC molar ratios were examined and optimized to get total glycerol conversion, highest selectivity, and yield of glycerol carbonate. Synthesized glycerol carbonate was characterized by ^{1}H and ^{13}C NMR spectra and gas chromatography analysis.

5.2 Catalyst synthesis process

Nickel nitrate hexahydrate [$Ni(NO_3)_2.6H_2O$], Magnesium nitrate hexahydrate, [$Mg(NO_3)_2.6H_2O$] , glycerol and dimethyl carbonate (DMC,99%), ammonia solution (25 wt. % in H_2O) were obtained from Merck scientific Ltd, Mumbai India. The chemicals used for this experimental work were of analytical grade. MgNi catalysts with various molar ratios (Mg/Ni=1:1, 2:1, 3:1) were synthesized taking respective stoichiometric amount by the co-precipitation route. For the synthesis of different catalyst, the required amount of Mg $(NO_3)_2 6H_2O$ and Ni $(NO_3)_2$ $.6H_2O$ were dissolved separately in deionized water. The prepared solutions were mixed in a beaker and undergo continuous stirring at room temperature for 6 to 7 h at 50°C with dropwise addition of liquid ammonia solution to maintain the pH 10-12. After proper precipitation occurred that was filtered and washed

with deionized water many times for obtaining the desired catalyst, the synthesized catalyst was kept in an oven at 110°C for 10-12 h and calcined in a muffle furnace at 700°C to get the designed catalyst. The prepared catalysts were specified according to molar ratio and calcinations temperature as m:n NiMg –T where m and n indicate the molar ratio of nickel and magnesium respectively, T refers to calcination temperature. All the synthesized catalysts with different molar ratios of Ni and Mg like 1:1, 1:2, 1:3 respectively was tested for glycerol carbonate synthesis and the catalyst providing highest conversion of glycerol at optimized reaction condition was characterized by several techniques like TGA-DSC, XRD, SEM-EDX etc.



Scheme 5.1 Synthetic method for NiMgOx catalyst via Co-precipitation route

5.3 Characterization of catalyst

5.3.1 TGA-DSC study

The thermal behavior of synthesized 3:1 Mg Ni catalyst was studied and the thermal stability curve is displayed in fig 5.1. From the graph it was noticed the synthesized catalyst was quite stable up to 100°C and the weight loss of the compound occurred gradually, the first state of weight loss of the catalyst was quite low i.e. approximately 4.76% which might be due to evaporation of adsorbed atmospheric water within the temperature range 100-200°C and similarly the second phase of the weight loss of catalyst at 200-300°C associated with decomposition of metal hydroxides to metal oxide or removal of crystalized water present in synthesized catalyst [136]. The final phase of the weight loss of the catalyst at temperature range 500-600°C might be due to decomposition of any basic nitrate or basic carbonates present in the synthesized catalyst. At 600°C, there was no further weight loss of the sample and the thermo gravimetric curve became parallel which might be due to complete decomposition of precursor to mixed metal oxides [137-139]. The further evaluation of thermal behavior of un calcined catalyst was provided by DSC analysis and it was endothermic at high temperature of 400°C due to decomposition of basic nitrate or hydroxyl carbonates present in the precursors.

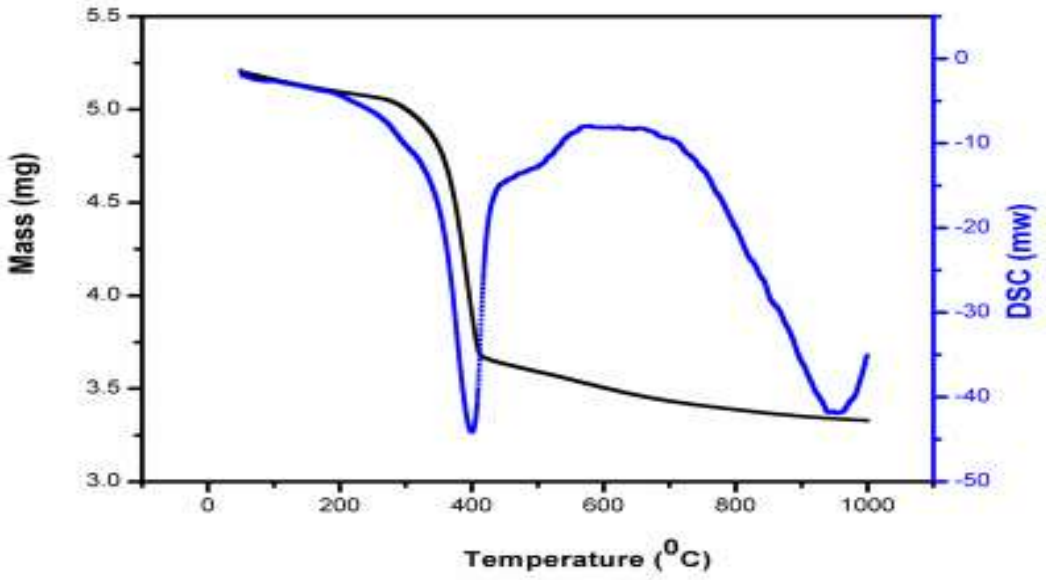


Fig 5 .1. TGA -DSC curve of uncalcined 3:1 Mg Ni catalyst

5.3.2 X-ray diffraction pattern

The XRD pattern of synthesized mixed oxide catalyst calcined at 700°C for 5h is shown in fig 5.2 (a). All the diffraction patterns were matched with JCPDS card number- 240712 and found to be face-centered cubic lattice (FCC). The average crystallite size of the catalyst was calculated from the most intense peak (200) using Scherrer equation (2 θ = 43.4°) and 26 nm for 3:1 Mg Ni catalyst. From the XRD graph, it was noticed the reflection patterns about 2θ = 37.2° ,43.2°, 62.6°, 74.7° and 79.2° were mainly due to MgO crystal planes intensity (JCPDS -650476) and the patterns at 2θ = 37.3°, 43.5°,62.8°,74.9° and 79.3° were the corresponding peaks of NiO (JCPDS-895881) phases with same miller indices values of MgO. Being all the positions of reflection pattern of NiO and MgO were of similar and shifted to merge into single peak as a result, the distinct patterns were detected at 2θ values of 37.3°, 43.4°, 62.58°, 75.10°, 79.07°with miller indices (111), (200), (220) ,(311) and (222) respectively which also attributed to the formation of solid $NiMgO_x$[140–142].

The consequence of loading of Mg^{2+} content on Ni^{2+} ion was well explored by XRD pattern within 2θ range 34- 44°and depicted in fig 5.2(b). There was a shifting of diffracted peaks close to miller indices values of (111) and (200) to lower diffraction angles with an increase in Mg^{2+} content as compared to pure NiO which might be due to the substitution of Ni^{2+} by Mg^{2+} in the NiO crystal lattice. The reflection pattern of mixed Ni Mg lattice was gradually intensified with a steady increase in Mg content as compared to pure NiO.

Fig 5.2(c) illustrated the diffraction pattern of 3:1 MgNi catalyst calcined at different temperatures. With rise in temperature from 500-700°C, the diffraction pattern were intensified gradually because of growth of MgO crystals at high temperature and at 800°C the diffraction patterns were de-intensified which might be due to sintering of metal oxides at this temperature which also affected the crystal phase of the synthesized catalyst indicating the optimum calcination temperature was about 700°C [143-144].

The influence of calcinations temperature on the reflection patterns of 3:1 MgNi catalyst was also studied and shown in fig 5.2(d).The miller indices like (111), (200) at 2θ value 37.2° and 43.4° respectively at 500°C started shifting to higher diffraction angle maximized to 37.8° and 43.6°at 700°C. Such type of phenomenon might be due to strong interaction of NiO with MgO at high temperature, since at low temperature there was no certain bonding between both the metal atoms occurred and asymmetry in $NiMgO_x$ lattice was investigated. The asymmetry in Ni-Mg lattice accounts for lower diffraction angle at lower temperature 500°C which at 700°C returned back to the original position and highly intensified due to similar ion radii between Ni^{2+} and Mg^{2+} [145-147] . Beyond 700°C, the reflection patterns moved again towards lower diffraction angle and also less intensified indicating the optimum calcination temperature was 700°C for the designed catalyst.

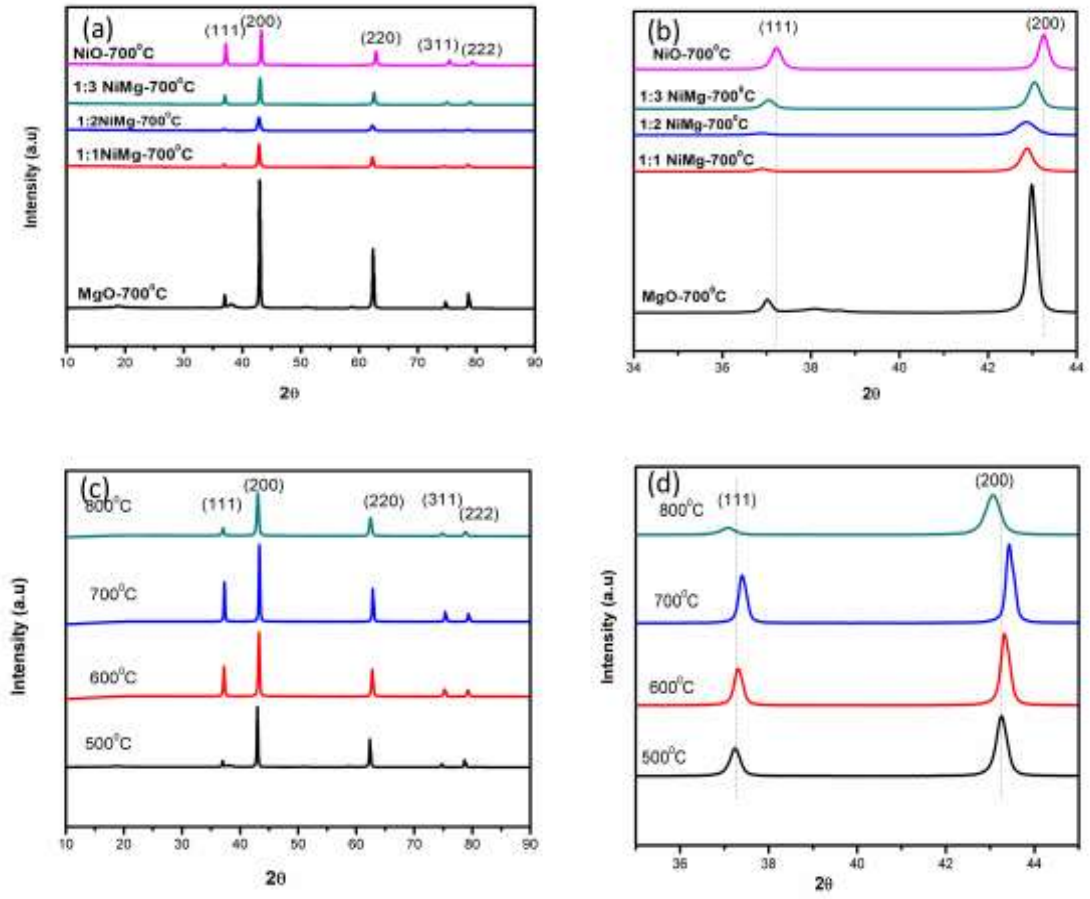


Fig 5. 2 XRD pattern of NiMgOx calcined at 700°C (a) 10-90˚, (b) Enlargement of patterns in the range of 34-44˚, (c) 3:1 Mg Ni catalyst calcined at different calcination temperature, (d) 3:1 Mg Ni calcined at different temperatures within 2 theta range 34-44˚

5.3.3 FT-IR spectra

The surface functional groups present in fresh 3:1MgNi catalyst, same catalyst after six times run in transesterification reaction of glycerol without calcinations and after calcined at 700˚C were studied through F-IR spectroscopy and shown in fig 5.3. The characteristics absorption bands were explained in the wave number range of 400- 4000 cm^{-1}.The very intense absorption band at 863cm^{-1} ascribed to stretching vibration of metal oxygen bonding (M –O bond) of freshly prepared catalyst which was broaden after sixth run in transesterification of glycerol and broadness of the absorption band reduced gradually

when calcined again at 700°C which might be due to the strengthening of M-O bond and evaporation of any adsorbed species [136,141]. The vibration band in the range of 1500-1250cm^{-1} (i.e.1460 cm^{-1}) highly responsible for bending vibration of O-H group of the physically adsorbed water molecule and after six times run in transesterification of glycerol it became doublet and broad which might be due to adsorption of any organic components along with water molecule and again calcined at 700°C the absorption band became sharper in nature[121,126] . Similarly, the band at 1034 cm^{-1} appeared in 3:1 MgNi catalyst after six time run in transesterification might be due to C-O stretching vibration of any organic moiety present in catalyst which became weaker and disappeared from the catalyst calcined at 700°C[148-151]. The broad band at 3430cm^{-1} was due to O-H stretching vibration of surface water molecule present in synthesized catalyst. The result obtained through FT-IR analysis matched well with the XRD analysis.

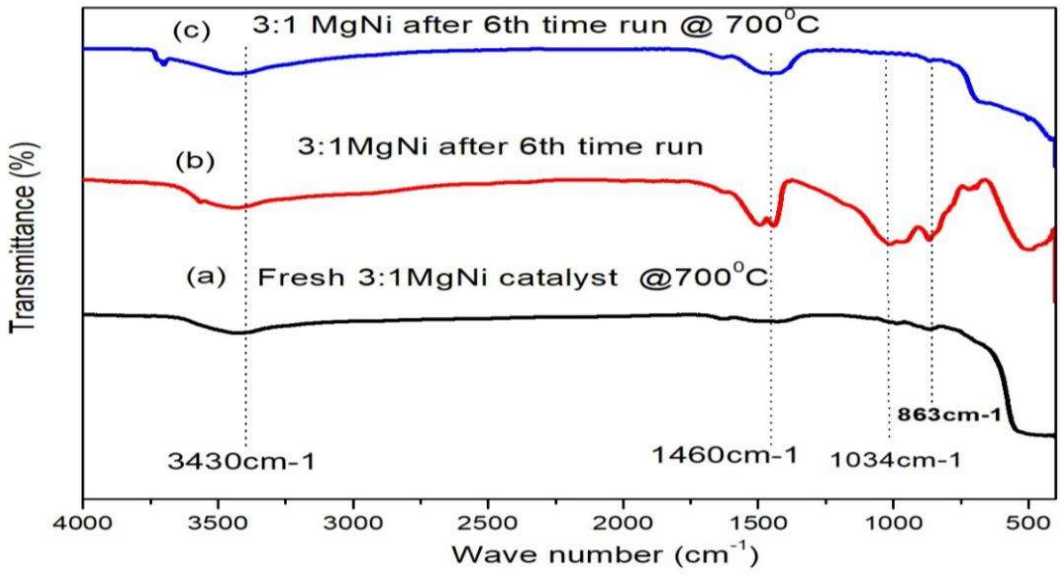


Fig 5.3. FT-IR spectra of (a) freshly prepared 3:1MgNi catalyst calcined at 700°C, (b) after 6th reuse cycle without calcinations, (c) after 6th reuse cycle and again calcined at 700°C.

5.3.4 SEM-EDX study

The morphology of synthesized 3:1 MgNi catalyst calcined at different temperatures were depicted in fig 5.4. The mixed oxide having variable grain type structure and with rise in calcination temperature the particle size of the mixed oxides increased gradually due to growth of bigger crystals. From the SEM image it was observed the particle size as well as porosity of catalyst gradually increased with rise in temperature from 500°C to 700°C and there was a compact network between the metal oxides calcined at 700°C which provided larger contact area between substrate and catalyst and influenced transesterification reaction. Above 700°C the particle size of 3:1 MgNi catalyst decreased which might be due to sintering of metals at high temperature indicating the optimum calcination temperature was about 700°C. The elemental composition of synthesized catalyst was verified by Energy dispersive X-ray spectroscopy (EDX) analysis. From the respective atomic and weight percentages it was revealed that the catalysts were composed of Ni, Mg and O with required atomic and weight percentage.

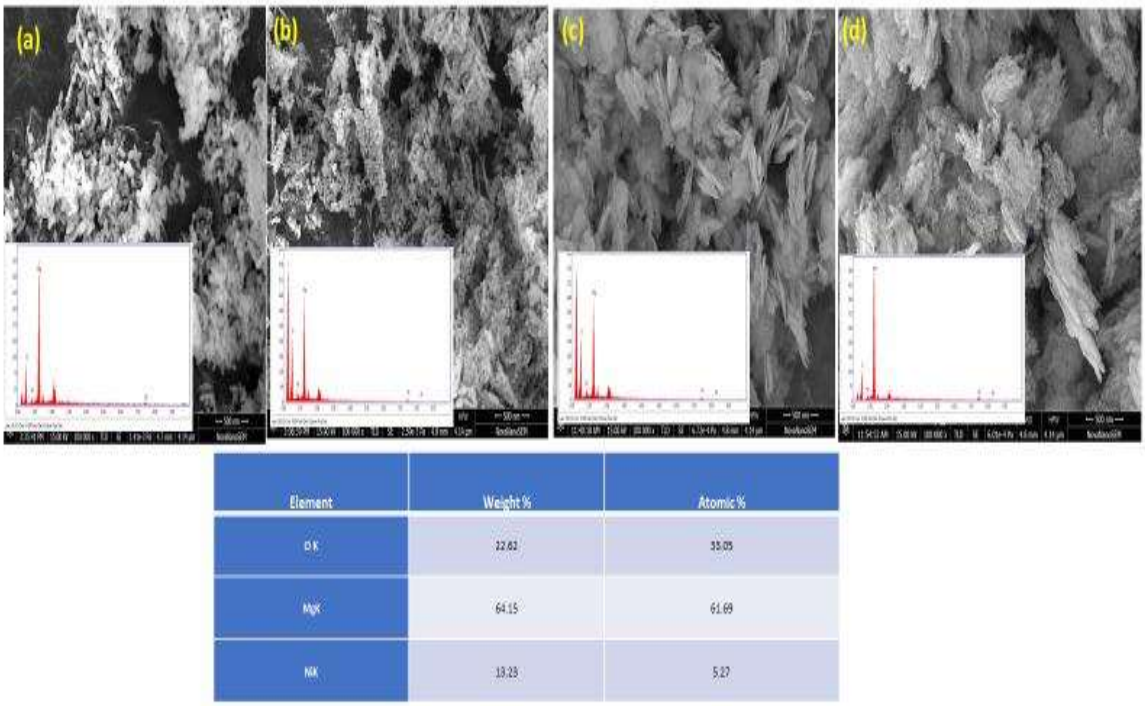


Fig 5.4 Surface morphology of synthesized (a)3:1 Mg Ni calcined at 500° C, (b) 3:1 Mg Ni at 600° C, (c) 3:1 Mg Ni at 700° C, (d) 3:1 Mg Ni at 800°C.

5.3.5 XPS spectra

The oxidation state and the surface chemical composition of the synthesized catalyst were studied by X-ray photoelectron spectroscopy (XPS) and portrayed in fig 5.5. The deconvolution of three characteristic peaks of O 1s spectrum which were asymmetric in nature at binding energies (BE) 530.12, 532.16, and 529.76 eV might be due to surface lattice oxygen with metals in mixed oxides, adsorbed oxygen species (O_{ads}) and water molecules bounded in the catalyst (H-O-H) respectively [152-154]. The main peak and the shake-up satellite peaks of 2p spectrum of nickel were compared with Ni^{2+} and Ni^{0}surface species which confirms the mixed oxidation state of nickel in the synthesized 3:1 Mg Ni catalyst. The main peak of Ni $2p_{3/2}$ at BE 855.56eV with a satellite peak at 6.4 eV higher binding energy corresponds to Ni^{2+} oxidation state, similarly the main

peak at binding energy 862.91eV associated with Ni $2p_{1/2}$ as well as shake up satellite peaks which confirmed the existence of mixed oxidation state of nickel atom [155–157]. From the XPS plot another peak at BE 1305.60 eV corresponds to 1s spectra of magnesium oxide confirming the presence of Mg^{2+} oxidation state.

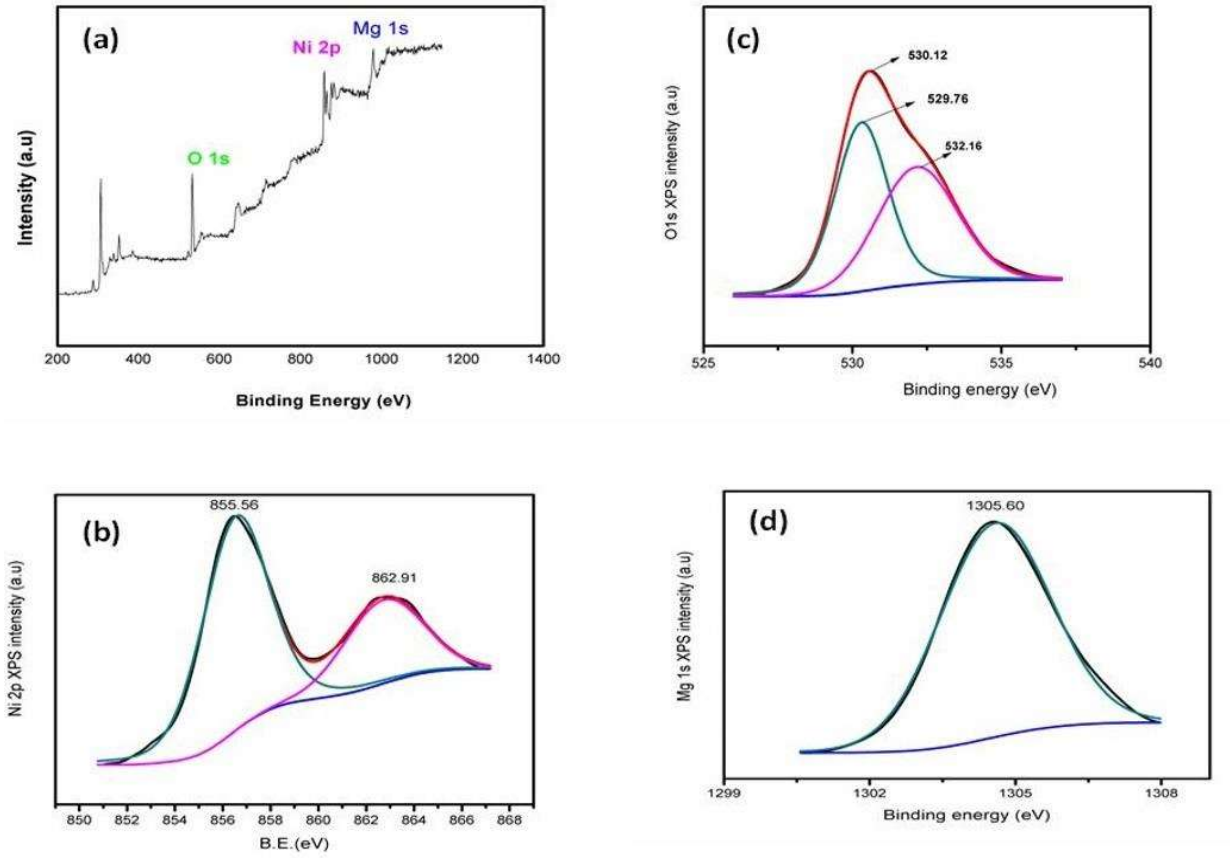


Fig 5.5. XPS spectra of (a) 3:1 MgNi Catalyst (b) Ni 2p spectra (c) O 1s spectra (d) Mg 1s spectra.

5.3.6 Surface area and basicity study of designed catalysts

The textured equities like (surface area, pore-volume, and pore diameter) of the designed mixed metal oxides were measured through BET surface area analyser and the results obtained are provided in Table 5.1. The synthesized catalysts had mesoporous texture as the pore size lies in the range of 2-50 nm. The 3:1 Mg Ni catalyst had lowest specific surface area in comparison to other synthesized 2:1 Mg Ni and 1:1 Mg Ni catalyst calcined at same

temperature 700°C, that might be due to partial covering of pores by the deposited MgO molecules. The N_2 adsorption desorption isotherm of 3:1 MgNi catalyst calcined at different temperature is shown in fig 5.6. The isotherm curve of 3:1 MgNi catalyst became larger and sharper with increase in temperature from 600° to 700°C might be due to removal of water molecules and carbonate groups from the catalyst at this temperature. At 800°C the isotherm curve started decreasing due to agglomeration of particles at high temperature. The significant loss in surface area might be due to the sintering of fine crystals which leads to agglomeration in pore walls and partial occlusion of the pores [158–161] . The basic strength of synthesized Ni Mg mixed metal oxide catalyst calcined at 700°C was calculated by using Hammet indicator benzene carboxylic acid titration technique. The basic strength of the prepared catalysts was studied by simple acid-base titration technique using Hammet indicators like phenolphthalein ($H_- = 9.3$), 2,4- dinitro aniline ($H_- = 15.0$), 4-nitro aniline ($H_- = 18.4$) and aniline ($H_- = 27.0$). The total basicity was calculated in the range of lowest basic strength, $H_- < 7.2$ and represented in the Table 5.1.

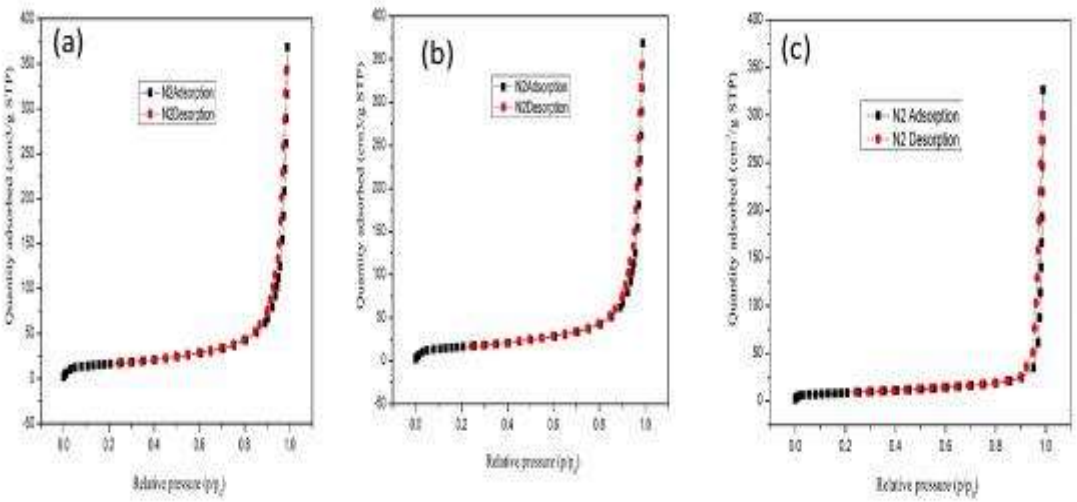


Fig 5.6 N_2 adsorption /desorption isotherm of (a) 3:1 MgNi calcined at 600°C, (b) 3:1 MgNi calcined at 700°C (c) 3:1 MgNi catalyst calcined at 800°C

Table 5.1: Properties of Synthesized 1:1, 1:2, 1:3 Ni Mg catalysts

Properties	1:1 NiMg	1:2 NiMg	1:3 NiMg
Total basic site density ($mmolg^{-1}$)	0.23	0.42	0.51
Total acidic site density($mmolg^{-1}$)	3.01	2.60	2.47
Basic site per unit surface area	7.26	8.83	14.98
Crystallite size (nm)	14	18	26
Lattice constant d(nm)	0.28	0.31	0.30
BET surface area (m^2g^{-1})	34	31	27
Specific pore volume (cm^3g^{-1})	0.14	0.09	0.07
Average pore diameter(nm)	23	17	14

5.4 Evaluation of catalyst for glycerol carbonate synthesis

The transesterification reaction of glycerol was performed in a 100 ml round bottom flask connected to a reflux condenser. Glycerol and dimethyl carbonate were taken as 1:4 molar ratio with 4wt % loading of catalyst (based on glycerol weight in gram used in the reaction mixture) and stirred at 500 rpm at 90°C temperature for 90 min. After completion of reaction process, the product mixture was centrifuged to obtain the desired liquid sample and the catalyst used in the reaction mixture. The reused catalyst was washed with methanol three to four times for further utilization in transesterification reaction. Then the obtained liquid sample was undergone for gas chromatography analysis using capillary column HP-5 with flame ionization detector (FID) in split injection mode. Initially, the column temperature was set at 75°C with 2 min of holding time, then the temperature gradually increased at a rate of 20°C min^{-1} up to 225°C. The temperature of injector and detector were kept at 260°C and 280°C respectively during product analysis. The transesterification reaction was repeated up to 5 to 7 times and conversion percentage of glycerol was

calculated. Similarly, the conversion percentage of glycerol was calculated also using ^{1}H NMR spectra using the equation as

$$\text{Glycerol conv (\%)} = \frac{b'CH}{b\,CH + b'CH} \times 100 \qquad (5.1)$$

Where b'_{CH} is the integration of methine proton of GLC.

b_{CH} is the methine proton of glycerol

Both ^{1}H and ^{13}C NMR spectra of synthesized glycerol carbonate is depicted in fig 5.7 as below. After the separation of catalyst from the reaction mixture the by-product methanol obtained was evaporated by rotary evaporator and the synthesized glycerol carbonate undergone for proton as well as carbon 13 analysis using Bruker Ascend TM 500MHz spectrometer. Dimethyl Sulfoxide–d_6 was used as a solvent and internal reference. The proton NMR spectra of synthesized glycerol carbonate is shown in fig 5.7 (a) and found to be different types of multiplet peaks at different chemical shift values responsible for protons present in the compound. The multiplet peak of chemical shift value of 4.81 ppm is mainly responsible for obtaining the conversion percentage of glycerol-to-glycerol carbonate and can be calculated by the integration area of proton NMR signal at chemical shift of 4.81ppm for methine proton of glycerol carbonate and integration area value for chemical shift of 3.43ppm for methine proton(-CH-) of glycerol left in reaction mixture. From the figure 5.7(b) shown for ^{13} C spectra of glycerol carbonate it was observed that there were four characteristic peaks of glycerol carbonate due to presence of four different carbon atoms. The peak at 155.55ppm corresponded to carbonate carbon peak of glycerol carbonate, including this other three peaks were also observed at 77.09 ppm, 66.46ppm and 60.66ppm for O-CH_2, O-CH and CH_2OH carbon atoms respectively. Another multiplet peak around 40ppm was observed mainly due to the solvent dimethyl sulfoxide. Since there was not complete conversion of glycerol in transesterification processes the other two peaks

at chemical shift values 63.42 and 72.30 corresponded to CH-OH and CH_2-OH carbon atoms of glycerol present in reaction mixture [130-131].

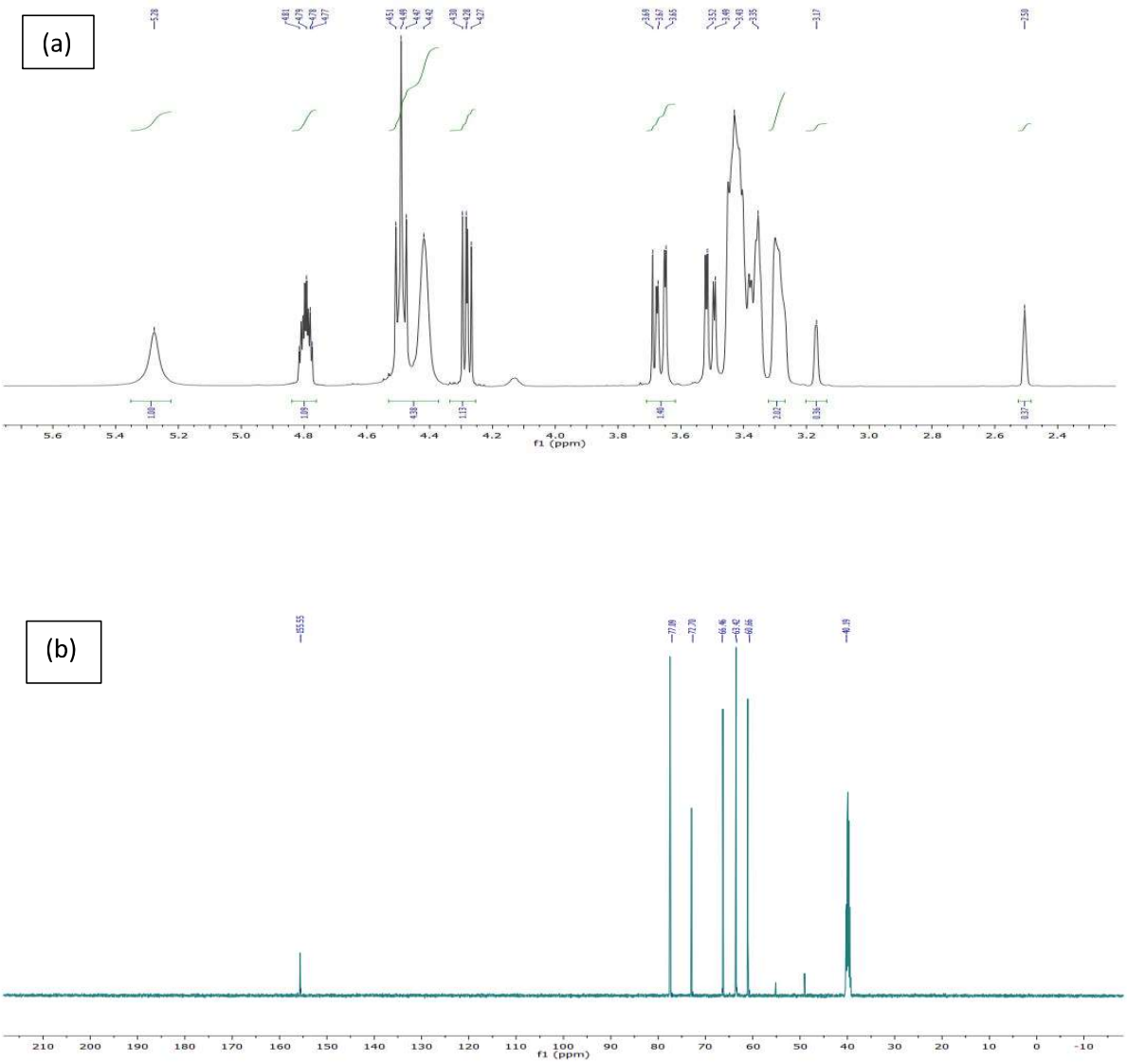
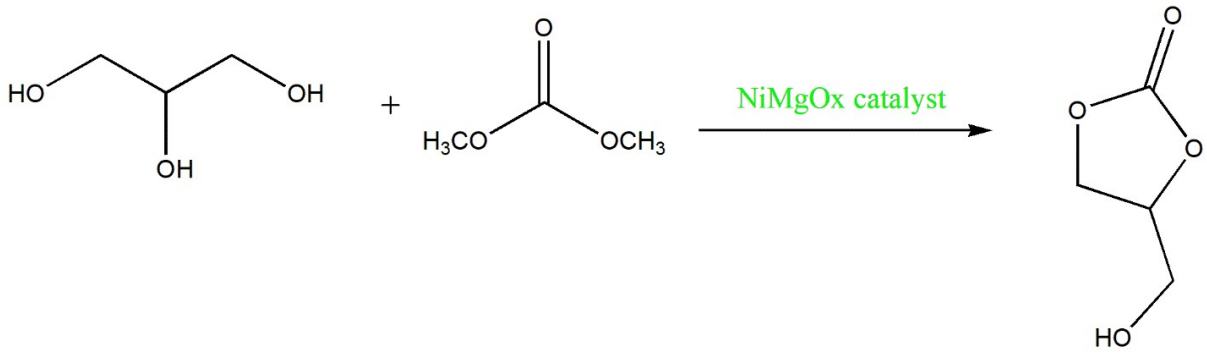


Fig 5.7 (a) ^{1}H NMR spectra, (b) ^{13}C NMR spectra of glycerol carbonate at optimized reaction conditions

5.5 Detailed study of reaction mechanism in transesterification of glycerol

A plausible reaction pathway for glycerol transesterification using dimethyl carbonate (DMC) and NiMgOx catalyst is presented in fig 5.8. The basic sites of catalyst react with glycerol to abstract H^+ ion and forms glyceroxide anion ($C_3H_7O_3^-$). The Lewis acidic site present in NiMgOx enhanced the positive character of carbonyl carbon of DMC through co-ordination with carbonyl oxygen, as a result nucleophilic reaction occurred between glyceroxide anion and DMC. The glyceroxide anion reacts with dimethyl carbonate to form hydroxyl alkyl carbonate which is highly unstable intermediate and undergoes cyclization to form glycerol carbonate and methanol as side product [70,115].



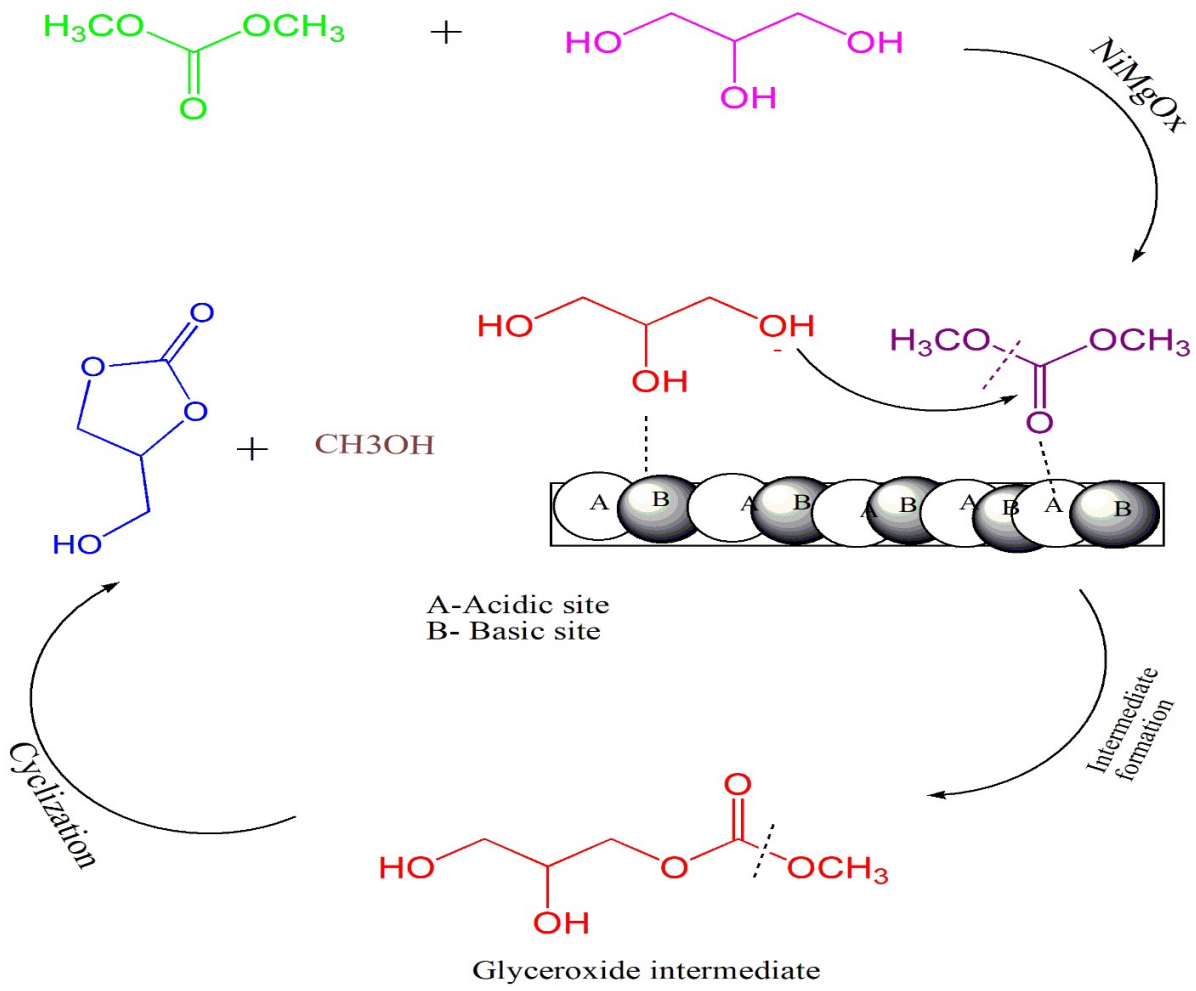


Fig 5.8. Plausible reaction mechanism of transesterification of glycerol catalysed by $NiMgO_x$ catalyst

5.6 Optimization study of reaction parameters

5.6.1 Effect of catalyst loading percentage

The transesterification reaction of glycerol was highly influenced by the catalyst loading percentage and presented in fig 5.9 (a). It was noticed that with an increase in loading percentage of 3:1 MgNi catalyst from 1 to 4 wt. % the conversion of glycerol increased from 62% to 97% and GLC yield also increased up to 82%, such types of trends in rise of both yield and conversion percentage might be due to the increase in overall basic sites based on the increase of catalyst amount. Above 4 wt.% of 3:1 MgNi catalyst the glycerol

conversion percentage became constant and the catalytic efficiency started decreasing due to agglomeration at a higher mass of catalyst as well as increased external mass transfer resistance in the reaction mixture and also blockage of pores of active sites of catalysts [162-164].

5.6.2 Effect of DMC to glycerol molar ratio

The influence of DMC to glycerol molar ratio in transesterification reaction was examined in the range of 1:1 - 6:1 sequentially keeping all the reaction parameters constant (i.e. T= 90°C ,time = 90 min, 4 wt.% 3:1 MgNi catalyst) and shown in fig 5.9 (b) ,with rise in DMC to glycerol molar ratio the conversion as well as yield of glycerol and GLC increased up to 97% and 82% respectively at 4:1 molar ratio of DMC: Glycerol by shifting the reaction equilibrium towards right, above a certain value of DMC to glycerol molar ratio the conversion percentage of glycerol and yield of glycerol carbonate did not increase significantly due to counterbalancing effect. Another factor responsible for lowering the rate of reaction was with rise in molar ratio of DMC to glycerol above 4:1 causes decrease in the concentration of glycerol as a result rate of transesterification reaction falls down [165-167].

5.6.3 Effect of reaction temperature

The temperature being one of the important factors in glycerol transesterification process was optimized and shown in fig 5.9 (c). The maximum temperature for the reaction process where the highest yield of glycerol carbonate was obtained is 90°C since this temperature provided sufficient kinetic energy to reactants for accelerating transesterification of glycerol. It was also observed that with rise in temperature from 30°C to 90°C, both the conversion and yield percentages of product mixture increased which gradually started decreasing above 90°C, might be due to dehydrogenation and

condensation reactions of by product methanol [168] .Another factor mainly responsible for lowering the conversion percentage of glycerol was at high temperature the evaporation of dimethyl carbonate takes place (above 90°C) as a result proper transesterification of glycerol did not occur since the boiling point of DMC about 90°C causing three phases in the reaction mixture : glycerol in liquid phase ,catalyst in solid phase and at high temperature the formation of glycidol occurred due to decarboxylation of glycerol carbonate which also further facilitate polyglycerol synthesis by base catalysed ring opening polymerization[169-170].

5.6.4 Effect of reaction time

The rate of transesterification of glycerol in presence of 3:1 Mg Ni catalyst was highly influenced by time and shown in fig 5.9 (d). The conversion of glycerol and yield of glycerol carbonate increased up to 97% and 82% respectively at 90 min under the same reaction conditions. Above 90 min, the glycerol conversion as well as yield of glycerol carbonate were not further increased indicating that the reaction attains equilibrium in that period and further increase of reaction time there was no significant increase of conversion percentage of glycerol[171-172].

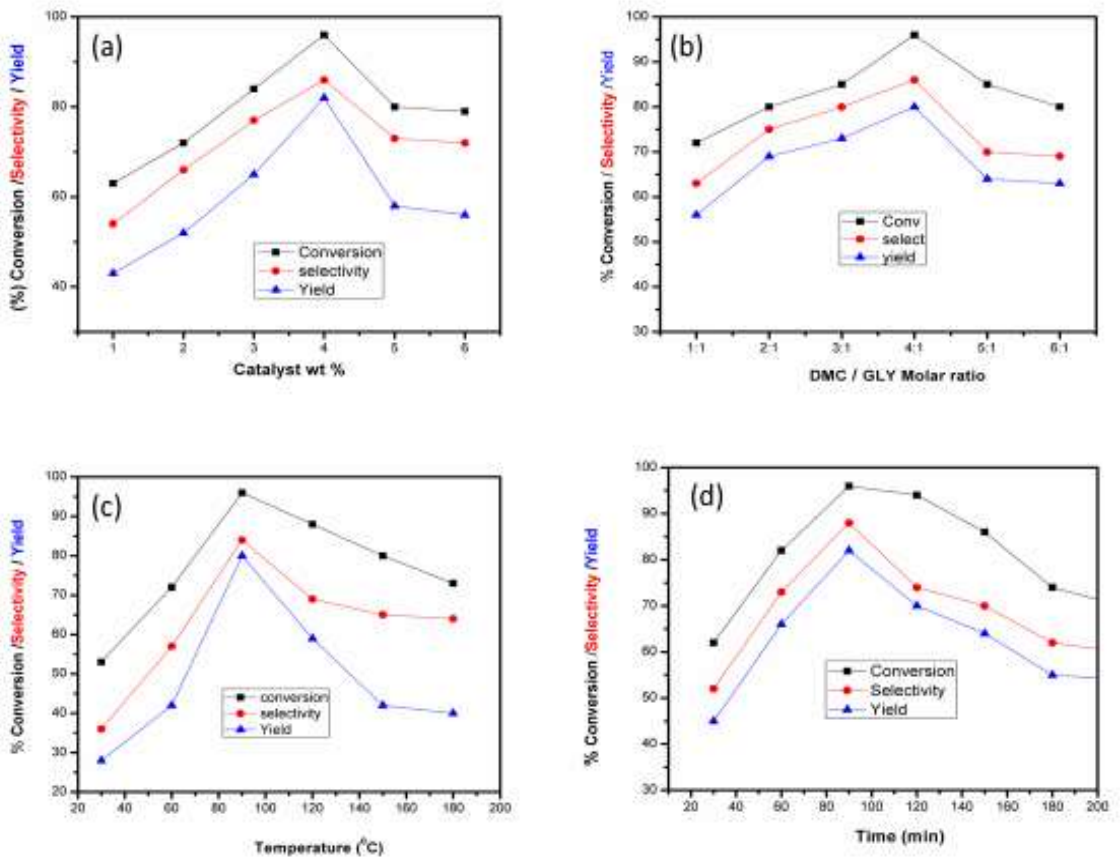


Fig 5.9. Effect of reaction parameters on transesterification of glycerol with DMC using 3:1 Mg Ni catalyst, (a) effect of catalyst loading percentage, (b) effect of DMC/Gly molar ratio, (c) effect of reaction temperature, (d) effect of reaction time

5.6.5 Reusability study of catalyst

The reusability test of 3:1 MgNi catalyst was carried out and the obtained graph was shown in fig 5.10. The catalyst was washed with methanol 3 to 4 times, dried in an oven at 110°C and calcined at 700°C for 6h after each consecutive reactions for further use in the transesterification reaction. It was observed that the catalytic activity as well as glycerol conversion slightly decreased with increasing reuse cycle, up to fifth cycle the conversion and yield of glycerol and glycerol carbonate were 82% and 73% respectively, at sixth cycle there was a sudden decrease in activity of catalyst which was due to reduction of basic

strength of catalyst from 15 < H_ < 18.4 to 9.3 < H_ < 15 for sixth reused catalyst confirmed by Hammet indicator test. In order to know the reason behind the loss of activity of catalyst, several characterization techniques like XRD, SEM-EDX and FT-IR were performed and respective figures are shown in fig5. 11 and fig 5.3. From XRD result it was clear there was decrease in intensity of diffraction peaks of sixth reused 3:1MgNi catalyst as compared to fresh catalyst and slight change of crystallinity after sixth runs in transesterification reaction. The EDX analysis of sixth reused 3:1 Mg Ni catalyst suggested there was decrease in content of Mg from 54.1% to 42.7 % and Ni from 13% to 9.4% respectively which might be due to leaching of elements during the reaction [173–175]. The SEM-image of sixth reused 3:1 MgNi catalyst confirmed there was severe agglomeration of particles taken place which deactivate the active site of catalyst.

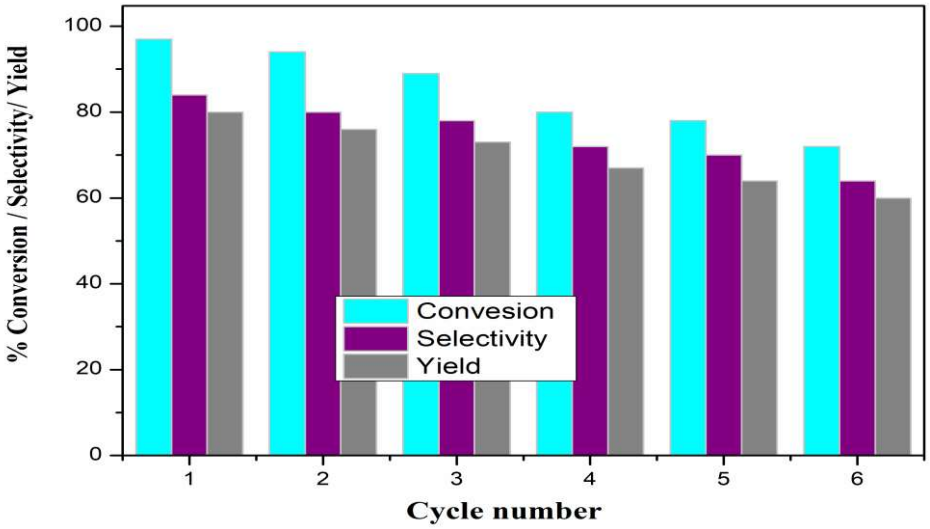
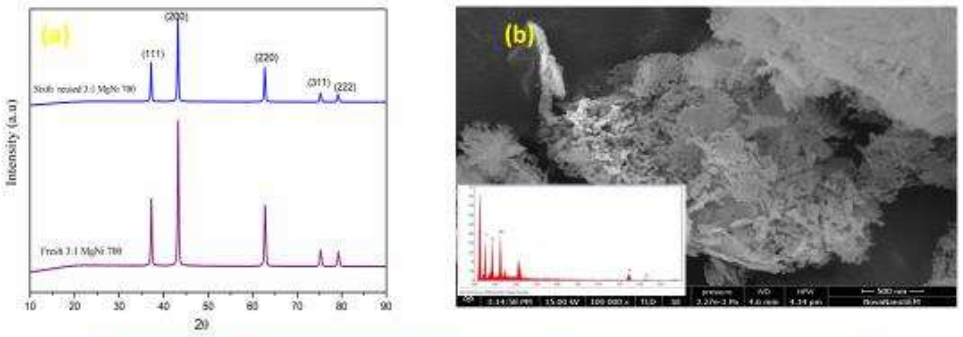


Fig 5.10. Catalyst reusability in glycerol carbonate synthesis at optimized reaction condition i.e., reaction temperature of 90°C, catalyst dose of 4 wt%, glycerol to DMC molar ratio 1:4, reaction time of 90 min.



(c) Elements	C	O	Mg	Ni
3:1 MgNi @700	10.4	22.5	54.1	13.0
Sixth reuse 3:1MgNi @700	13.8	34.1	42.7	9.4

Fig 5.11 Comparison of (a) XRD profile of fresh and Six times reused 3 :1 MgNi catalyst calcined at 700°C (b) SEM-image of sixth reused 3:1 MgNi calcined at 700°C (c) EDX analysis of fresh and sixth reused 3:1 MgNi at 700°C

5.7 Conclusion

Glycerol carbonate being a green and highly demanded industrial chemical was synthesized in our laboratory using a cheaper and greener heterogeneous NiMgOx catalyst. A series of catalysts were prepared with various molar ratios like 1:1, 2:1 and 3:1 Mg Ni and their activity and stability towards glycerol carbonate synthesis was studied. Depending on the activity of synthesized catalysts, 3:1 MgNi catalyst was found to be best catalyst for glycerol transesterification reaction at optimized reaction conditions. From the experiment, it was confirmed that higher basic strength of 3:1 Mg Ni is one of the factors for maximum conversion of glycerol (97%) since the reaction followed base catalyzed reaction. several

characterization techniques like XRD, FT-IR spectroscopy and XPS confirmed the existence of bimetallic mixed oxides at desired ratios. The catalyst was highly stable up to fifth run obtaining 76% conversion of glycerol and after 5^{th} cycle the conversion of glycerol reduced consequently due to rapid deactivation of basic sites of catalyst. Overall NiMgOx catalyst is a cost effective, simple and highly active catalyst for glycerol carbonate production. Further application of glycerol carbonate in the synthesis of various organic compounds having medicinal properties like hydroxy methyl-2-oxazolidinones can be extended in future.