

## Research Article

# Transformation of Biodiesel waste Glycerol to Value added Glycerol Carbonate

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## Abstract

Glycerol carbonate (GLC) is a promising and fascinating chemical of glycerol, which extensively used in pharmaceutical, cosmetic, and food industries as well as green solvent in chemical industries. GLC can be derived from glycerol, which is a waste of biodiesel production process and dimethyl carbonate using various homogeneous and heterogeneous catalysts by transesterification reaction. This review comprises the catalytic activity of various catalysts such as alkaline catalysts, Ionic liquid catalysts, double layered metal hydroxide catalysts and waste products as catalyst for synthesis of glycerol carbonate and optimization of reaction parameters. It was observed that heterogeneous catalysts mainly double layered mixed metal oxide catalysts are competent catalyst for production of glycerol carbonate with improved yield. Utilization of glycerol to fascinated green products minimizes the gluts of biodiesel and oleochemical industry and cleans environment. Henceforth these processes implement value addition to glycerol, reduce the cost and economic of biodiesel production.

**Keywords:** Glycerol, Dimethyl carbonate, Heterogeneous catalyst, Transesterification, Glycerol carbonate

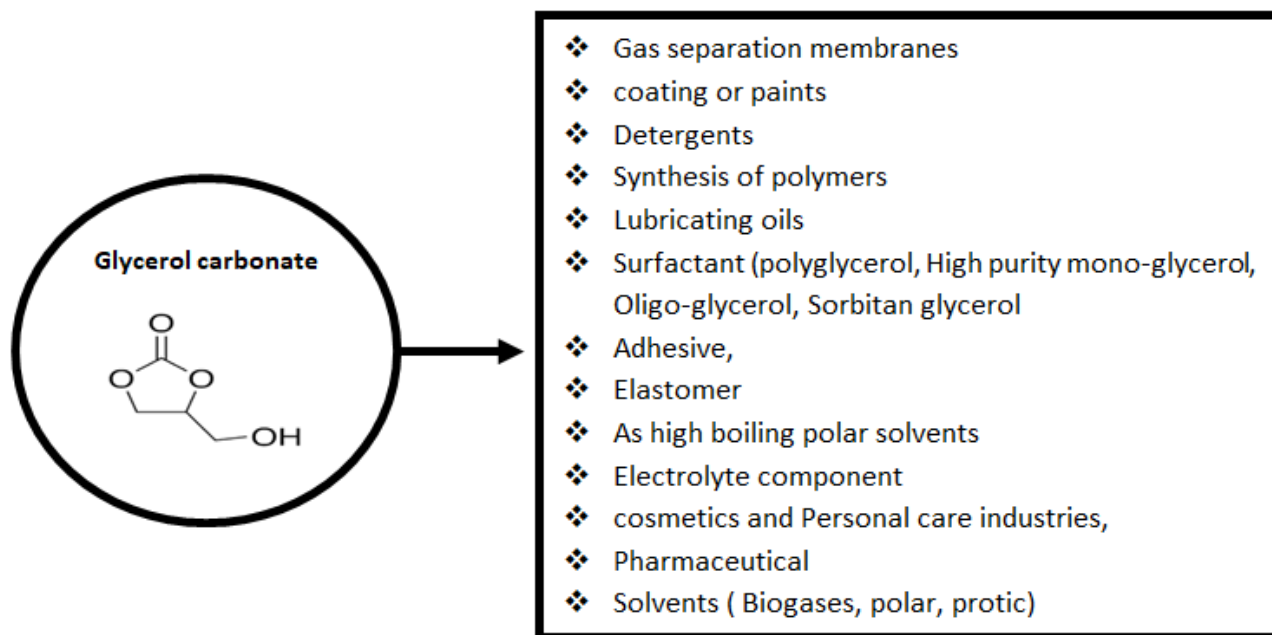
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## Introduction

Energy plays a strategic role for social and economic growth of any country. It serves as sustainable, affordable and secure resource for economic development purpose and might be a stipulation for sustainable development [1]. Nowadays, energy progress from biobased fuels to inexhaustible and sustainable vitality resources attracted much attention. The potentials of other energy resources like biofuels, wind, hydro, solar and geothermal provide the stable key to current global energy challenges [2-6]. Currently overuses of energy supply are unsustainable due to environment intention like global warming which is predominantly associated with enhancement energy consumption. Vitality adequacy and security is a major factor being developed since it gives basic contributions to financial advancement that offer essential types of assistance for socioeconomic development to date and provide the significance services to boost standard of life at regional, sub-national and national levels [7-8]. In recent years, plant based fuels and oils have been explored much to reduce the oil imports as well as to support the agricultural industries or other sectors for biofuels production from agricultural products. It has been several advantages in term of sustainability like gas emission, reduced pollutant, biodegradability, diversity and increases energy, also for economic security purpose [9]. Recently, there is vast synthesis of commercialized biodiesel from vegetables oils of non-edible variety as well as from animal fats and which have been directly employed in motor vehicles as whole (100% Biodiesel) or blends [10-14]. Biodiesel (BD) is produced by the transesterification of fatty oils with lower chain alcohols like methanol or ethanol [15-16]. During biodiesel production process, 10% w/w of glycerol is generated as an undesired by-product [17-23], which interpreted that about 2300 million liters glycerol produced in upcoming year 2025. Subsequently, the expanding worldwide interest for BD has come about in oversupplied glycerol, which has bad impacts on biodiesel industry. Pure glycerol could be a most precious chemical with thousand of opportunities for brand latest industrial application [24]. Glycerol has various applications in many fields like pharmaceutical, cosmeceuticals, food industries and used in cough syrups, toothpaste, allergen immune-therapies, mouthwashes, skin or hair care products, toiletries and glycerin soaps. Glycerol has also several medical applications; pure glycerol is used for treatment of bedsores, cuts, bites, rashes, burns and Nitroglycerin is used for treatment of cardiovascular disease [25, 26]. As a result the proper utilization of crude glycerol produced in biodiesel industries is most essential and now a-days researchers are focusing on value addition of glycerol [27]. Recently many researchers have explored several modern chemical pathways to maintain the sustainability in Glycerol and BD industry for the efficient modification of glycerol to fascinating chemicals. GLC (4-hydroxymethyl-1, 3-dioxolan-2-one / Glycerol carbonate) is a most valuable and important compound of glycerol [28] that draws in noteworthy logical and mechanical

enthusiasm because of its low poisonousness, low combustibility, biodegradability, good flammability and also good water solubility. Highly flammable and halogenated solvents like ethoxyethane were limited utilized in synthesis of GLC. There is still requirement to replace non-protic and polar solvents like N-methyl-pyrrolidine-2-one and N, N-dimethylformamide with more acceptable alternatives [29]. During this context, glycerol transformation into high worth included synthetics is incredibly important. GLC being one of the interesting fine chemicals derived from glycerol and utilized in various industries is represented in **Figure 1**. It is widely used in gas separation membranes, coating and painting, detergents, synthesis of polymers like polycarbonates, polyamides, polyurethanes and polyesters, lubricating oils, surfactant, adhesive, elastomer, as high boiling polar solvents, electrolyte component and it is also utilized in plastics, cosmetics textile, pharmaceutical industries[26].



**Figure 1** Applications of Glycerol Carbonate [26]

The present review focused on synthesis of GLC from Glycerol and Dimethyl carbonate by utilizing various homogeneous and heterogeneous catalysts and the impact of various reaction boundaries, for example, catalyst stacking, molar proportion of DMC/glycerol, temperature and reaction time was investigated for optimized reaction condition.

## Materials and Methods

Glycerol and DMC plays major role in the synthesis of GLC by transesterification reaction in presence of metal doped base catalysts. Conventional wetness impregnation technique was followed to prepare different metal doped base catalysts. Transesterification reaction was carried out in a 100ml round bottom flask connected to a reflux condenser. The reaction was carried out taking different stoichiometric proportion of Glycerol and dimethyl carbonate using different weight percentage loading of catalyst (based on glycerol used in the reaction mixture). The reaction mixture was stirred in a magnetic stirrer vigorously at 500rpm at 90 °C temperature for 1.5 hr. Then the product mixture was centrifuged to separate out the desired reaction product and the catalyst used in the reaction. The used catalyst was washed three to four times with methanol for reutilization in the transesterification reaction.

### *Synthetic pathway of glycerol carbonate*

GLC could be obtained by several methods including: addition of CO<sub>2</sub> to glycerol [30, 31], addition of glycerol to CO [32] and glycerolysis of urea [33]. However, GLC basically synthesized via direct synthetic route and indirect synthetic route. The synthetic route involves carbon monoxide and carbon dioxide, mightily attractive because it would be a way to eliminate the noxious greenhouse emission [34]. But, this route is not thermodynamically favored and vitality concentrated. There are also other indirect route which, includes the glycerolysis with urea, phosgenation and transesterification with carbonates such as dialkyl and alkyl carbonates (**Figure 2**). The indirect pathway having several features sort of a facile, minimal energy necessity for synthesis of GLC and also it is non-thermodynamic limited pathway [34, 35, 36]. GLC could be synthesized via dialkylene carbonates and alkylene carbonates function

of carbonate resources, is a noticeable offer for GLC synthesis. They are an attractive choice as compared to toxic phosgene and glycerolysis of urea because this reaction required vacuum (40–50 mbar) [37] to remove continuously ammonia from gaseous phase to proceed reaction. Dimethyl carbonate (DMC) is widely adopted for GLC synthesis, because it is cheap and exhibit low toxicity, higher efficiency at mild reaction phases which esteem higher yield GLC from substantial glycerol [38-44]. The transesterification reaction of glycerol with dimethyl carbonate generates one particle of glycerol carbonate and two particles of methanol, where the equivalent dimethyl carbonate can be delivered from carbon dioxide and methanol [45]. The reaction of transesterification is considered as equilibrium reversible reaction. Hence, continuous removal of methanol as well as excess DMC is required, to obtain the sublimable GLC yield [46, 47]. Furthermore, with using ethylene and propylene carbonates are overcomes steps encountered in purification and separation using DMC. Thermodynamic stability and thickness of glycerol is significant factors, which require generally vitality concentrated conditions to accomplish the cleavages and initiation of reaction [34]. Thus, catalysts plays vital role to proliferate the transesterification reaction at mild reaction conditions.

Researchers have been explored and reported that GLC can be synthesized using heterogeneous, homogenous and also enzyme catalysts. They demonstrated that selectivity of glycerol carbonate synthesis largely depend on catalyst type and also on other reaction parameters tuning to avoid formation of undesired by product. Some researchers reported acetylation and carboxylation of biodiesel-based glycerol with dimethyl carbonate and acetic acid to synthesize GLC and acetyl esters of glycerol. They also investigated optimization of reaction conditions, catalyst activity or deactivation and kinetics of reaction for effective synthesis of GLC [34, 35]. The transesterification reaction of glycerol with DMC is thermodynamically compatible and can be employed to prepare GLC. In this paper comparative analysis of different catalysts used for GLC synthesis by transesterification reaction of glycerol with DMC is extensively discussed.

## Result and Discussion

GLC synthesis by transesterification reaction using glycerol with DMC, mostly depends on optimal reaction conditions and alkaline catalysts [48-50] Thus, the higher yield of GLC successfully obtained by application of several kind of catalysts to sustain the reaction [34, 51, 52]. Heterogeneous catalysts are considered as best catalyst over traditional homogeneous alkaline catalysts due to its easy separation, reusability and less corrosive nature. Homogenous catalysts such as KOH,  $K_2CO_3$ , NaOH successfully accustomed synthesize GLC via transesterification reaction using glycerol with DMC [53] but homogenous catalysts are environmental hazardous.

### *Enzymes as catalyst*

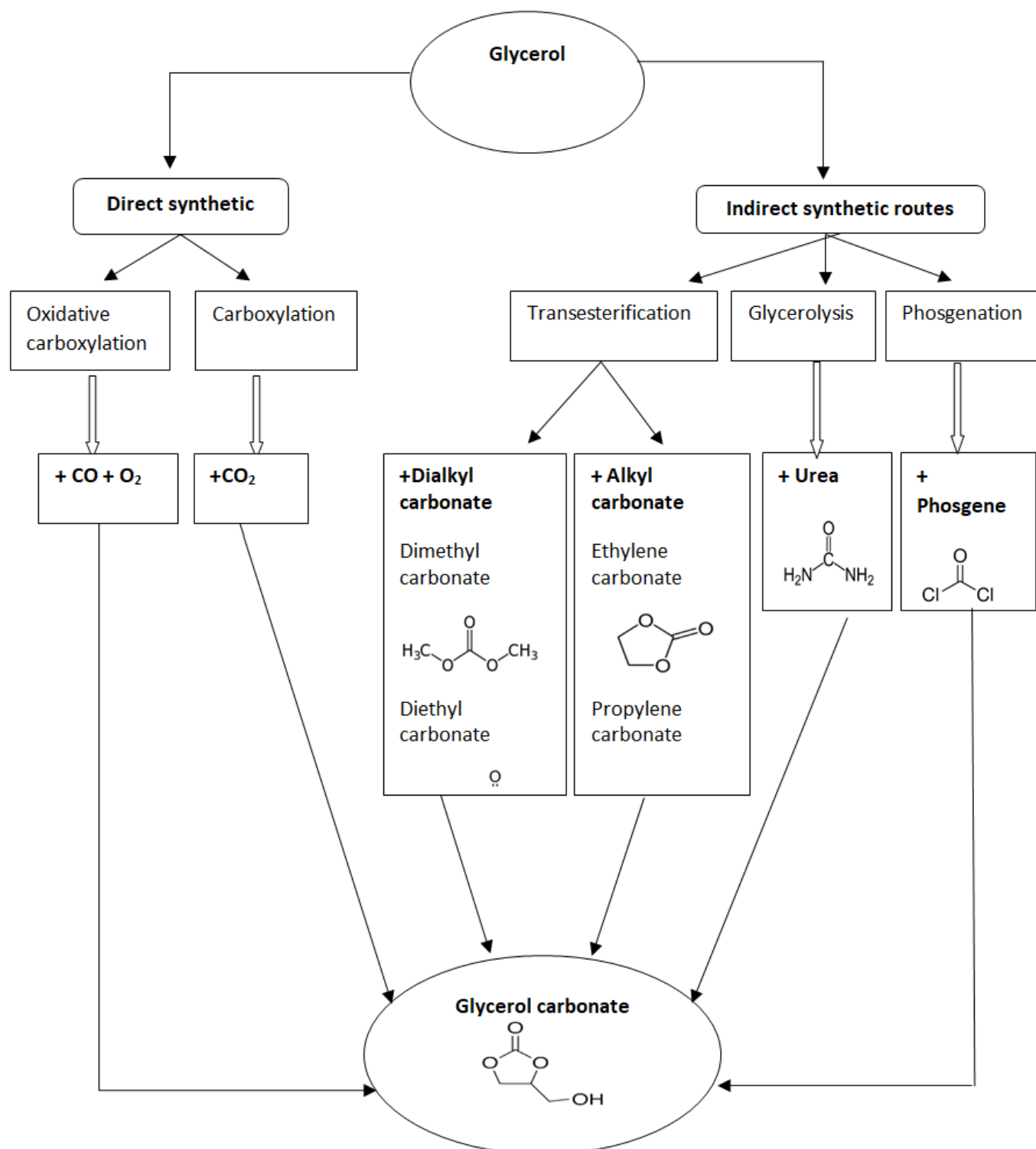
Enzymes were employed to reduce the employment of hazardous catalyst which posed by homogenous catalysts and improve the green production of GLC synthesize using DMC with glycerol [54]. Enzymes required prolonged reaction time about 6- 48 hrs with less yield of GLC about 36% [34]. Some enzymes are still expensive and required higher glycerol /DMC molar ratio yet as lower reaction temperature (<60 °C) [55, 56]. To overcome fast enzyme deactivation and diminish their price, enzymes bonded on magnetic nanoparticle have been used. Tudorache et al. [56] revealed that a low stacking of lipase on magnetic nano particle increases the activity of enzyme catalyst due to uniform dispersion under mild reaction conditions which possess selectivity of GLC about 90% with glycerol conversion of 36%. Therefore, the lipase based on nano-magnetic catalyst could be undergo 15 times cycles of reuse without deactivation but still achieve lower yield of GLC relatively with reaction time about 6 hours.

Du and co-workers [53] reported using lipase enzyme moored on magnetic silica nano-flowers comparably achieve a higher yield of GLC. They used surfactant and molecular sieve to extend miscibility and adsorb water respectively. Using surfactant and 0.2 g molecular sieve, conversion of glycerol is about 94.24% and GLC yield is 88.66% at glycerol/DMC molar ratio of about 1:20 at 50 °C for 24 hours latent period and seven times reusability of catalyst. Hence, researchers have given more attention towards heterogeneous catalysts to avoid the drawbacks of enzymes and homogeneous catalysts. The catalytic activity of heterogeneous catalyst is comparably greater than homogeneous catalyst (GLC yield >90%) [57-60].

### *Alkaline Catalyst*

Transesterification reaction is mainly associated with proton abstraction to provide the reactive intermediate which is favoured by alkaline catalysts [45]. During the synthesis of GLC in presence of alkaline-base catalyst, cyclization reaction occurs with reactive intermediate and the carbonyl group of DMC. Basic catalysts achieved high conversion than the acidic catalysis during the transesterification reaction so base catalyst seems more promising than the acid

catalysis [61]. On comparing various basic solids like, MgO, BaO, SrCO<sub>3</sub>, these catalyst becomes more active because of having intrinsic basicity and MgO catalyst shows more activity which prepared by hydration followed by calcination billboard material [62].



**Figure 2** Glycerol carbonates synthesis by various techniques using glycerol [26]

The alkaline catalyst such as CaCO<sub>3</sub>, CaO are required to perform transesterification reaction by using DMC but this catalyst shows less reactivity after use [63]. A number of other catalysts such as Mg/Al/Zr mixed oxides [64], KF modified hydroxyapatite [65] and triethylamine [66] or Lipases [67] even are used during the transesterification reaction. Homogeneous catalysts such as K<sub>2</sub>CO<sub>3</sub>, NaOH, H<sub>2</sub>SO<sub>4</sub>, KOH, p-toluenesulfonic acid, DBU (1,8-diazabicyclo[5.4.0]undec-7-ene), and DABCO (1,4-diazabicyclo [2.2.2] octane) were exhibited to be highly deedful for this transesterification [68-70]. These type catalysts having some practical problems during the reaction in terms

of reuses and cannot easily separate but they exhibit high catalytic activity. There is decomposition of GLC occurred due to existence of homogeneous catalysts during the distillation separation of GLC and glycerol [71]. Finally, catalysts based on calcium such as  $Mg_{1-x}Ca_xO_2$  and CaO,  $CaCO_3$ ,  $CaO/Al_2O_3$ , or  $Ca(OCH_3)_2$  employed as a brand new solid catalyst for GLC synthesis from DMC and glycerol by transesterification reaction that showed high catalytic activity. However, this calcium based catalysts easily separate, recovered and formed calcium-based homogenous highly active species during the transesterification reaction [53, 72-73]. They offered reasonably high selectivity for glycerol carbonate and high catalytic activity of glycerol conversion. Various inorganic or organic base catalysts like DBU, DABCO, ionic liquids, alkali metal, alkaline earth metals, mixed metals oxide, abundant materials or wastes like disposable diapers, dolomites, ash, eggshells, steel slags, fishmeal and gypsum are employed with considerable performance for GLC synthesis selectivity [44,74-77].

### *Ionic liquids (ILs) as catalyst*

Ionic liquids (ILs) have been utilized as catalysts during the synthesis of GLC. Ionic liquids are solvents, which absolutely contain ions that are fluid at ambient temperature [46, 78]. Particularly, basic ionic liquid contains amide, quaternary salts of imidazolium, ammonium similarly as quadine, which has been successfully employed in GLC production to catalyzing the transesterification reaction of glycerol with DMC [46, 79-80]. Ionic liquid shows several types of disadvantages such as separation after the completion of reaction, ion mobility, anion basicity and various types of undesired by products could be generated when they are employed in a reaction [81].

### *Layered double hydroxides (LDH) as catalyst*

GLC also successfully synthesized by using heterogeneous catalysts such as mixed metals oxides that are based on, magnesium, potassium hydrotalcite, sodium and calcium-based catalysts. Hydrotalcites are layered double hydroxide (LDH) compounds, with the general formula  $[M^{2+}_{1-x}M^{3+}_x(OH)_2]^{x+}[A^{p-}_{x/p}]^{x+}.mH_2O$ , where charge balancing anion represented by  $A^p$  and divalent and trivalent cations respectively represented by  $M^{2+}$  and  $M^{3+}$ [82]. Different mixed oxides are obtained from LDHs which could be easily recovered after rehydration. LDH have memory impact i.e., on thermal treatment over 400 °, it changes into separate blended oxides, that could be recouped after rehydration [83]. The blended oxides obtained from LDH decay have a generally high surface territory, which is thermally stable and solid Lewis base. Hence, it showed intermediate GLC yield and conversion of glycerol was successfully improved by using LDH decomposed mixed oxides as catalyst in GLC synthesis [84, 85]. There is limited interaction of primitive LDH or disintegrated blended oxides due to their low solubility. This restriction affects the utilization of harsh response conditions and larger reaction time of 4-54 h is required for completion of glycerol transformation even at temperature over 120 °C [86]. To stay away from brutal response conditions, solvents for example, DMF (*N,N*-dimethyl formamide) have been utilized to accomplish a 75% GLC yield at mild reaction temperature of 100 °C in 1hr [87].

The equivalently low synergist execution of unblemished or decayed LDH is notably attributed to low solubility and essential quality of magnesium. Basicity of LDH can be increased by the centralization of metal cations consolidated over LDH structures, for example copper [42],  $NaAlO_2$  [39],  $Mg/Al/Zr$  [88] and transition metals have been used to increase the hydrotalcites basicity to improve GLC yield [65]. Moreover, CaO and catalysts of mixed calcium are promising heterogeneous catalysts for the glycerol transesterification reaction with DMC to prepare GLC. The economical abundant nature of CaO and high basicity, makes it attractive catalyst for transesterification reaction of glycerol with DMC [80].

Appreciable GLC yield of >90% obtained from calcined calcium oxide over 900 °C when DMC is utilized in overabundance of equal stoichiometric ratio [89]. However, calcined CaO is much unstable in glycerol and highly strong to deactivate by atmospheric  $CO_2$ . Thus, catalytic activity of CaO drastically diminished during the recycling performance by CaO [53, 89, 90]. Some metals, for example- lanthanum (La), potassium (K), sodium (Na) and magnesium (Mg) have been utilized to stabilize CaO [72, 91, 92]. These metals exhibit synergistic and dispersion effect on CaO and increases its basicity as well as stability. Above study also concluded that use of alkaline and alkali earth metals stabilized CaO and the reusability of catalyst increases to more than four cycles. Therefore, intense exhausting chemical substances are fully substituted by abundant materials or wastes with requisite elemental compositions to achieve sustainability of GLC synthesis.

### *Wastes as catalyst*

Algoufi and co-workers achieved improved GLC yield of above 95% by using calcined dolomite and K-zeolites, a reusable catalyst originated from waste coal fly ash [93, 94]. Our research group at IIT BHU, used active metal doped

pond ash catalyst for GLC synthesis. Biochar of agricultural waste has been used as potential catalysts for transesterification reaction of glycerol due to high surface area, low-cost and inflexible textural characteristics. About 99% of GLC yield was obtained using fishery waste with glycerol/DMC molar ratios of 2 at 85 °C for 90 min reaction time [95]. The fishery waste contains an admirable quantity of phosphorus, calcium, potassium, sodium, carbon, nitrogen and oxygen. Basicity of biochar and pore characteristics was enhanced by pyrolysis at a temperature of 500-700 °C, hence the reusability of biochar catalyst increases and after fifth time of reuse yield of GLC is 93% [96]. Okoye et al. [97] investigated the new pathways for GLC synthesis using ash of empty fruit from palm which shows the improved effect due to calcination. They revealed that by increasing temperature of calcination from 300 °C to 600 °C, the crystalline phase of ash was modified from  $KAlSiO_4$  to  $K_2Mg(SiO_4)$  and basicity increases, which showed improved GLC yield of more than 95% with 3 molar ratios of DMC/glycerol at 90 °C in reaction time of 45 min. The stable calcined ash could be reused for four times without rapid deactivation. Similarly high surface area lithium doped zeolite was produced from palm oil ash and used as a catalyst for transformation of glycerol to value added GLC [98]. Lithium doping enhanced the surface basicity and 100% glycerol conversion was occurred with 98% GLC yield at DMC/glycerol molar proportion of 2, temperature of 70 °C and reaction time of 90 min. After doping with Li, the crystallinity of zeolite framework remains preserved and the catalyst can be reused for five times without immense deactivation [45]. The activity of different catalysts, optimization reaction condition and GLC yield was compiled in **Table 1**.

**Table 1** Correlation of reaction conditions and execution of heterogeneous base catalysts in glycerol transesterification [26]

Catalysts	Solvent	Reaction conditions			Performance <sup>b</sup>	
		M.R	C.L <sup>a</sup> (wt or mol %)	Temp (°C)	R.T(h)	Y/C/S (%)
CaCO <sub>3</sub> <sup>d</sup>	–	5:1 (DMC)	10 mol%	75	1.5	Y=90.6
CaO <sup>d</sup>	–	5:1 (DMC)	6wt%	75	1.5	Y=91.1
CaO <sup>d</sup>	–	2:1 (DMC)	3mol%	75	0.5	Y=94
CaO	–	2:1 (DMC)	3mol%	75	0.5	Y=90.2
CaO	–	1:1 (DMC)	3mol%	60	2	Y=69
CaO	–	2:1 (EC)	0.5wt%	35	1	Y=83
CaO	–	2:1 (EC)	0.5wt%	35	0.25	Y=81
Calcium complex Ca(C <sub>3</sub> H <sub>7</sub> O <sub>3</sub> ) <sub>2</sub>	DMF	5:1 (DMC)	8 mol%	60	3	Y=95
MgO <sup>d</sup>	–	2:1 (DMC)	3mol%	75	3	Y=10.2
ZnO	–	2:1 (DMC)	3mol%	75	0.5	Y=0.5
Na <sub>2</sub> O	–	2:1 (DMC)	3mol%	75	0.5	Y=92.6
MgO	–	2:1 (EC)	7wt%	50	5	Y=78
Al/Mg hydrotalcite <sup>d</sup>	–	2:1 (EC)	7wt%	50	5	Y=82
Al/Mg hydrotalcite <sup>d</sup> (rehydrated)	–	2:1 (EC)	7wt%	50	5	Y=68
Al/Ca hydrotalcite <sup>d</sup>	–	2:1 (EC)	0.5wt%	35	1	Y=87
Mg1 + xCa1 _ xO2 mixed oxides	–	2:1 (DMC)	3wt%	70	1.5	Y=100
Mg/Al/Zr mixed oxides	–	5:1 (DMC)	0.1wt%	75	1.5	Y=94
Mg/Al hydrotalcite	DMF	5:1 (DMC)	54wt%	100	3	Y=82
Mg/Al hydrotalcite (rehydrated)	–	17:1(DEC)	16wt%	130	10	Y=65
Zeolite (NaY) <sup>d</sup>	Methanol	3:1 (DMC)	10wt%	70	4	Y=80
KF/hydroxyapatite	–	2:1 (DMC)	3wt%	78	0.83	Y=99
NaOH/c-Al <sub>2</sub> O <sub>3</sub>	–	2:1 (DMC)	3wt%	78	1	Y=97h

DEC = Diethyl carbonate, DMC = Dimethyl carbonate, EC = Ethylene carbonate  
M.R= molar ratio, TEMP= temperature, R.T=reaction time, C.L= catalysts loading  
Y = yield of glycerol carbonate, S = selectivity of glycerol carbonate, C = transformation of glycerol.  
<sup>a</sup>(Amount of catalyst/ measure of glycerol)×100%.  
<sup>b</sup>Y = (production of glycerol carbonate (g) / initial amount of glycerol (g)) ×100%,  
C = (initial glycerol – residual glycerol) / glycerol initial × 100%, S = Y/C  
<sup>d</sup>catalyst calcination for 3 h to overnight at 450°C ≤ T ≤ 6 900°C.

From the comparative analysis it was found that heterogeneous mixed metal oxide (Mg1 + xCa1 \_ xO<sub>2</sub>) catalyst showed high performance compared to other catalysts. Utilization of heterogeneous catalyst in transesterification reactions has been obliged by the reaction affecting boundaries as well as performance and stability. Optimization of parameters such as reaction temperature, response time, DMC to glycerol molar ratio and catalyst dose is critical for

acquiring high GLC yield. Among these parameters, particularly the reaction temperature as well as catalyst solidness is a significant element to study before commercialization of the GLC synthesis route.

## Conclusion

Glycerol carbonate is a valuable and important derivative of glycerol and is a green chemical. It has various industrial as well as other applications like accessory in manufacturing of polymeric materials such as polycarbonates, polyglycerol esters as well as polyurethanes, as intermediate in cosmetics or chemical synthesis, pharmaceuticals, detergents etc. The subsequent routes are analyzed to synthesize glycerol carbonate by various chemicals such as (a) reaction of glycerol with DMC or ethylene carbonate via transesterification; (b) reaction of glycerol with urea via carbonation or carbamoylation process; (c) reaction of CO<sub>2</sub> with 3-chloro-1,2- propanediol. As a consequence, these alternative routes have been selected for glycerol carbonate synthesis. Maximum yield of GLC can be obtained using different form of catalysts including hydrotalcites, lipase, ionic liquids and pure or mixed metal oxides via hydrotalcites and doped metal oxides during transesterification of glycerol. It is worth noting that production of GLC is closely passionate about the sort of catalyst applied in transesterification reaction.

Basic heterogeneous catalyst with proper basicity and fundamental quality can improve the GLC synthesis by transesterification reaction using glycerol and DMC. Hence, response temperature, catalyst stacking, DMC/glycerol molar proportion and response time influences the rate of GLC transformation. Subsequently, proper decision of these factors and tuning the response affecting variables will improve the GLC yield. Industrial manufacturing of GLC via transesterification reaction treated with ethylene carbonate or DMC with glycerol in presence of heterogeneous catalyst looked as if it would be the valuable and foremost convenient pathway due to their high activities, recycled abilities and easy recovery methods. Hence transformation of biodiesel waste, glycerol into valuable and fascinating chemical Glycerol carbonate will economize the biodiesel production process.

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