



## Preparation of Cr<sub>2</sub>O<sub>3</sub>, MnO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, NiO, CuO, and ZnO Oxides Using Their Glycine Complexes as Precursors for *In Situ* Thermal Decomposition

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**G**LYCINE (Gly) complexes play an important role in human life. Zn(II), Cu(II), Ni(II), Fe(III), Mn(II), and Cr(III) complexes were synthesized with glycine (Gly). These complexes were characterized by infrared spectroscopy (FT-IR). Based on the stoichiometric data, Gly form a chelate complex in a bidentate fashion at a 1:3 ratio with the trivalent metal ions [Cr(III), Fe(III)] and a 1:2 ratio with the divalent ones [Mn(II), Ni(II), Cu(II) and Zn(II)]. Metal oxides were derived from each of the Gly complexes using thermal decomposition at 800°C. FT-IR analysis of the resulting solids revealed their formation in pure form.

**Keywords:** Chelation, FT-IR, Glycine, Metal oxide, Thermal decomposition.

### Introduction

Recently, complexation between amino acids and transition metal ions has received much attention due to their broad and increasing number of applications, including as antibacterial agents with potent biological activity, particularly against *Escherichia coli* and *Staphylococcus aureus* [1-5]. These complexes have been utilized in a variety of biological processes [6-8], like electron transfer, oxygen conversion and oxidation, during which the enzymatic active site forms complexes with divalent metal ions [9-14]. A greater understanding of the chemical interaction between metal ions and the biologically active molecules is required to optimize certain aspects of their biocompatibility, particularly relevant when preparing biomaterials.

Studying the complexation of simple amino acids can inform the interpretation of more complex systems. Glycine (Gly; NH<sub>2</sub>CH<sub>2</sub>COOH; 75.07 g/mol) is the simplest amino acid, consisting of a single carbon bonded to carboxyl and amino groups, and is the only amino acid that does not have optical isomers. In the human body, Gly contributes to building muscle protein [15], absorbing calcium [16-18], producing antibodies [19-21], enzymes [22-24], and hormones [25, 26], and recovering from physical injury (e.g., incurred during surgery or from sports) [27-28]. On the other hand, in general, it has been observed during our recent articles, that the synthetic peptide chemistry are promising as biologically activate [29-44]. Therefore, it has become one of our most important trends, is to obtain different derivatives

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Received 8/12/2019; Accepted 29/1/2020

DOI: 10.21608/ejchem.2020.20679.2240

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of amino acids with transitional elements, recent research efforts have investigated the synthesis of metal oxides from complex precursors using thermal decomposition [45-48]. Herein, we report findings from the synthesis and IR-based characterization of Zn(II)-, Cu(II)-, Ni(II)-, Fe(III)-, Mn(II)-, and Cr(III)-Gly complexes (Fig. 1) and the preparation of ZnO, CuO, NiO, Fe<sub>2</sub>O<sub>3</sub>, MnO<sub>2</sub>, and Cr<sub>2</sub>O<sub>3</sub> using solid-state thermal decomposition of these complexes.

### Experimental Details

#### Chemistry

##### Chemicals and IR measurements

Gly, CrCl<sub>3</sub>, MnCl<sub>2</sub>, FeCl<sub>3</sub>.6H<sub>2</sub>O, NiCl<sub>2</sub>.6H<sub>2</sub>O, Cu(SO<sub>4</sub>)<sub>2</sub>.6H<sub>2</sub>O, and Zn(SO<sub>4</sub>)<sub>2</sub>.6H<sub>2</sub>O were used as received without no additional purification from Merck Chemical Company. A Bruker FT-IR spectrophotometer was used to collect the IR spectra (4000–400 cm<sup>-1</sup>) for the solid oxides, recorded as KBr pellets.

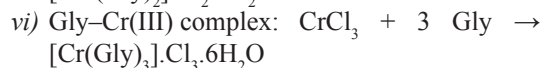
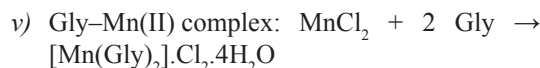
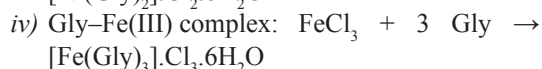
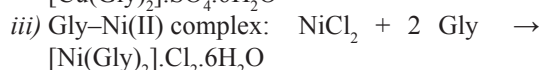
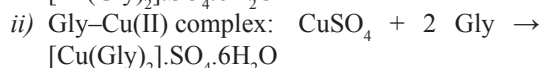
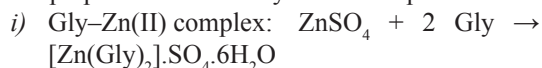
##### Synthesis of the precursors

Gly was dissolved in 25 mL of ethanol and mixed with each metal salts (CrCl<sub>3</sub>, MnCl<sub>2</sub>, FeCl<sub>3</sub>.6H<sub>2</sub>O, NiCl<sub>2</sub>.6H<sub>2</sub>O, CuSO<sub>4</sub>.6H<sub>2</sub>O and ZnSO<sub>4</sub>.6H<sub>2</sub>O) solution which were dissolved in 25 mL of distilled water to obtain the desired molar ratio: 1:2 (divalent metal salts) or 1:3 (trivalent metal ions) M:Gly. These mixtures were continuously stirring at 80 °C until approximately 50% of the volume evaporated. Then, all of the solutions were left about seven days at ambient temperature until precipitated. The ignition of the solid precipitations as precursors was carried out in an electrical oven at 800 °C for 3 hrs. Prior to this combustion reaction, small amounts of each solid precipitate were analyzed using FT-IR. The pathway of this procedure is summarized in a flow chart presented in Scheme 1 with NiCl<sub>2</sub>.6H<sub>2</sub>O as an example.

## Results and Discussion

#### Chemistry

The following chemical equations describe the preparation of the Gly-metal complexes:



The divalent metal ions, Zn(II), Cu(II), Ni(II), and Mn(II), reacted with the Gly ligand at the same molar ratio (1:2) but their coordination modes varied. The trivalent metal ions, Cr(III) and Fe(III), coordinated to the Gly ligand at a 1:3 molar ratio. Figure 2 contains the IR spectra of the synthesized Gly-metal complexes, where the proposed IR assignments for these complexes are listed in Table 1. These data indicated that extensive coupling occurred for several vibrations, which rendered qualitative deductions concerning the environment surrounding the metal ion challenging. However, the infrared spectra listed in Table 1 describe changes in the positions of some absorption ranges compared to those in Gly ligand alone, these changes suggest associated groups with these infrared bands when formatted into the metal ion. The most pronounced changes were observed in the bands corresponding to the amino and carboxylate groups. When in solution, the molecular form of the amino acid is pH-dependent. Zwitterion is an intermolecular neutralization reaction that leads to a salt-like ion. Scheme 2 shows the zwitterion structure of the Gly molecule in solution. The amino group of the Gly molecule can accept a hydrogen ion and become positively charged or lose a hydrogen ion and become negatively charged.

In the IR spectra of the synthesized complexes, the (NH<sub>3</sub>)<sup>+</sup> characteristic IR band corresponding to the ν<sub>2</sub> vibrations located at 3170 cm<sup>-1</sup> for the free Gly ligand was shifted and overlapped with the hydrogen bond from the crystalline water molecules. The band due to the NH<sub>3</sub><sup>+</sup> group ν(NH<sub>3</sub><sup>+</sup>) resonated at ~3124, 2950, and 2500 cm<sup>-1</sup> [8], which was very intense in the free Gly sample and appeared as a weak shoulder or disappeared in the IR spectra of the metal complexes. The stretching vibrations of the NH<sub>3</sub><sup>+</sup> group generated broad bands from 3100 to 2800 cm<sup>-1</sup> in the IR spectra of the complexes, while the stretching vibrations of the OH group gave a sharp band around 3200 cm<sup>-1</sup>. The band observed around ~1610 cm<sup>-1</sup> was assigned to ν<sub>as</sub>(COO<sup>-</sup>) vibrations, whereas that of the ν<sub>s</sub>(COO<sup>-</sup>) vibrations was located around ~1400 cm<sup>-1</sup>. These bands represent a Δν difference (asymmetric-symmetric) ranging from 161 to 248 cm<sup>-1</sup>, indicating that the carboxylate groups of the Gly ligands coordinated to the metal ions as

monodentate chelates [8]. The absorption bands observed from 500 to 400 cm<sup>-1</sup> in the IR spectra of the complexes were due to the stretching vibrations of the M–N and M–O frequencies. Taken together, all of these observations suggest that the Gly ligand coordinated to the metal ions via the oxygen atom of the carboxylate group and the nitrogen atom of the amino group.

The Gly complexes were thermally

decomposed in an electric furnace and the FT-IR spectra of the final residues were recorded (Fig. 3A-F) to illustrate the formation of Cr<sub>2</sub>O<sub>3</sub>, MnO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, NiO, CuO, and ZnO. The peaks at 614 and 534 cm<sup>-1</sup>, 595 cm<sup>-1</sup>, (522 and 431 cm<sup>-1</sup>, 430 cm<sup>-1</sup>, 422 cm<sup>-1</sup> and 425 cm<sup>-1</sup> in the FT-IR spectra of Cr<sub>2</sub>O<sub>3</sub>, MnO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, NiO, CuO, and ZnO, respectively (Fig. 3A-F) assigned to the M–O stretching frequencies [8].

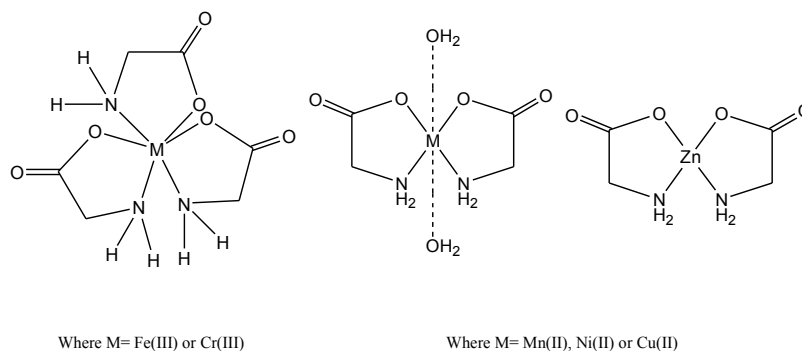
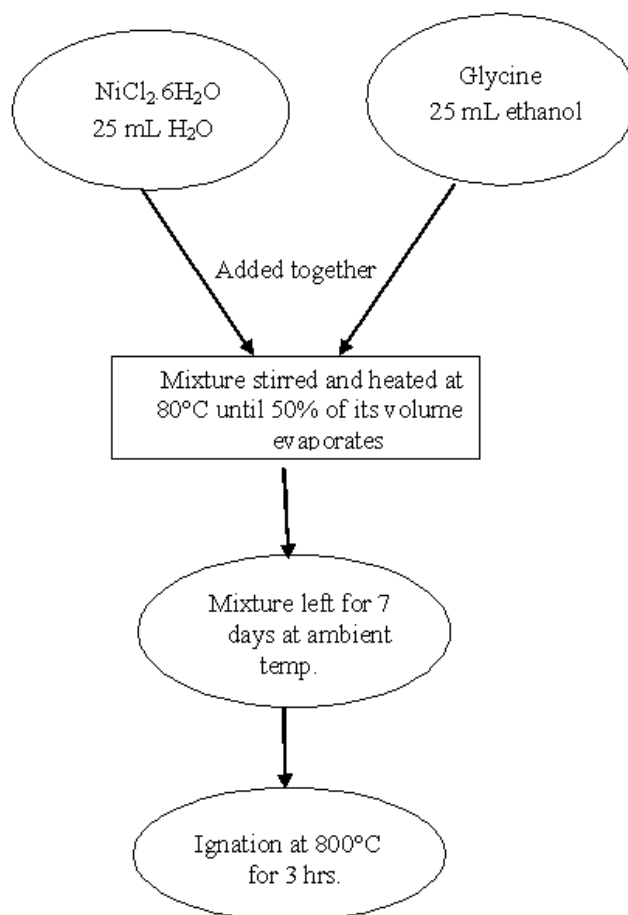


Fig. 1. Proposed chemical structures of Zn(II)-, Cu(II)-, Ni(II)-, Fe(III)-, Mn(II)-, and Cr(III)-Gly complexes.



Scheme 1. Preparation of the Gly-metal complexes and their corresponding metal oxides.

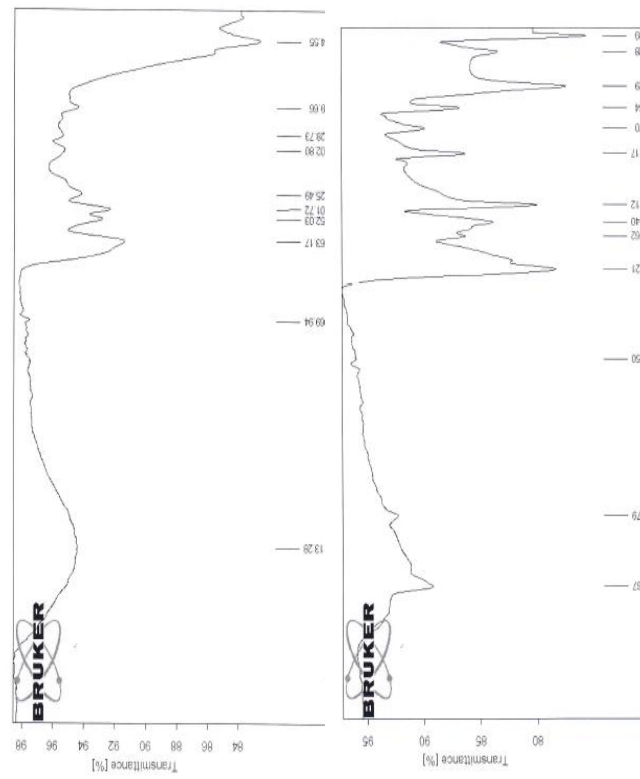


Fig. 2A

Fig. 2B

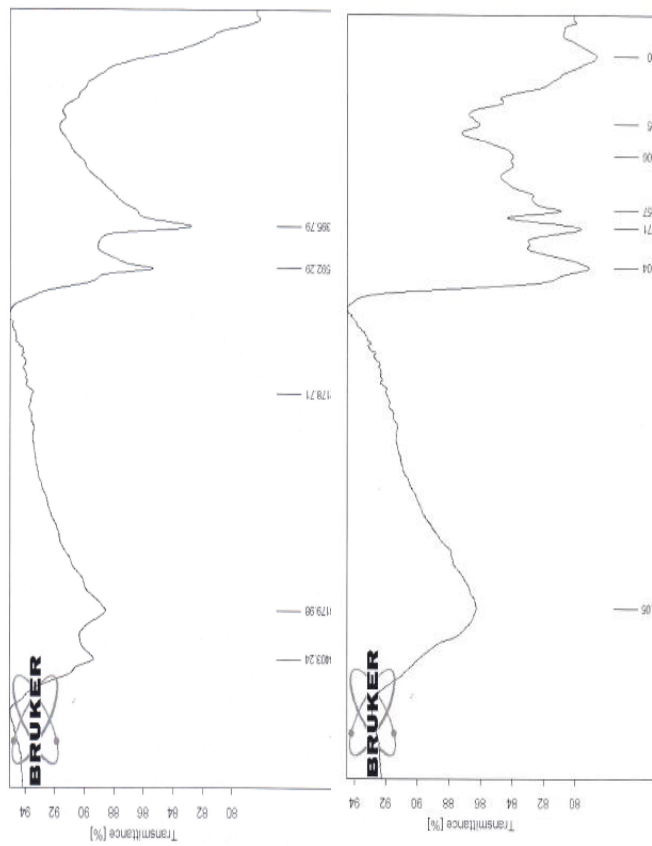


Fig. 2C

Fig. 2D

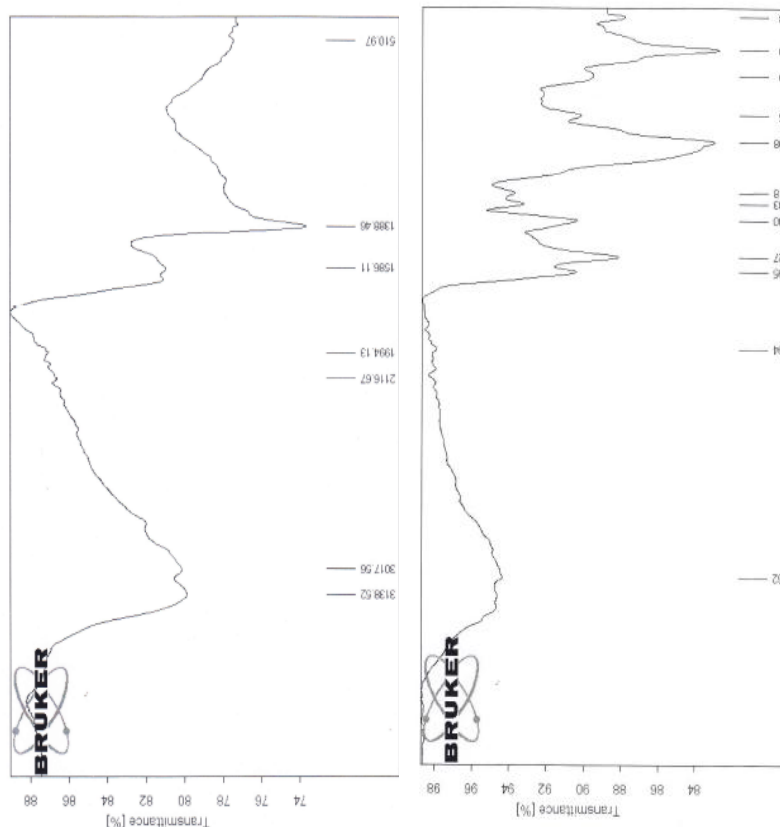


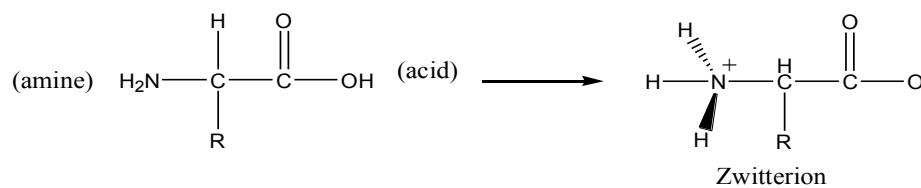
Fig. 2E

Fig. 2F

Fig. 2. FT-IR spectra of the Gly metal complexes (A): [Cr(Gly)<sub>3</sub>].Cl<sub>3</sub>.6H<sub>2</sub>O, (B): [Mn(Gly)<sub>2</sub>].Cl<sub>2</sub>.4H<sub>2</sub>O, (C): [Fe(Gly)<sub>3</sub>].Cl<sub>3</sub>.6H<sub>2</sub>O, (D): [Ni(Gly)<sub>2</sub>].Cl<sub>2</sub>.6H<sub>2</sub>O, (E): [Cu(Gly)<sub>2</sub>].SO<sub>4</sub>.6H<sub>2</sub>O and (F): [Zn(Gly)<sub>2</sub>].SO<sub>4</sub>.6H<sub>2</sub>O.

TABLE 1. FT-IR bands that differentiate between the free Gly ligand and corresponding metal complex.

Assignments (cm <sup>-1</sup> )	Compounds						
	Free Gly	Cr(III)	Mn(II)	Fe(III)	Ni(II)	Cu(II)	Zn(II)
OH str.	-	3113	3308	3403	3179	3138	3109
NH <sub>2</sub> str.	3170	-	-	3180	-	3017	-
COO <sup>-</sup> asym.	1611	1563	1652	1592	1586	1586	1650
δNH <sub>2</sub>	1507	-	1478	-	-	-	1578
COO <sup>-</sup> sym.	1414	1402	1405	1396	1401	1388	1404
		1325	1316		1316		1327
M-O str.	-	554	423	500	591	510	598
M-N str.	-	430	438	410	450	421	440



Scheme 2. The zwitterion structure of Gly (R is H).

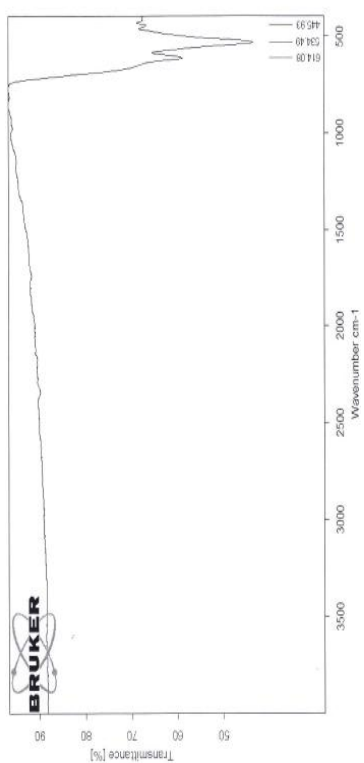


Fig. 3A

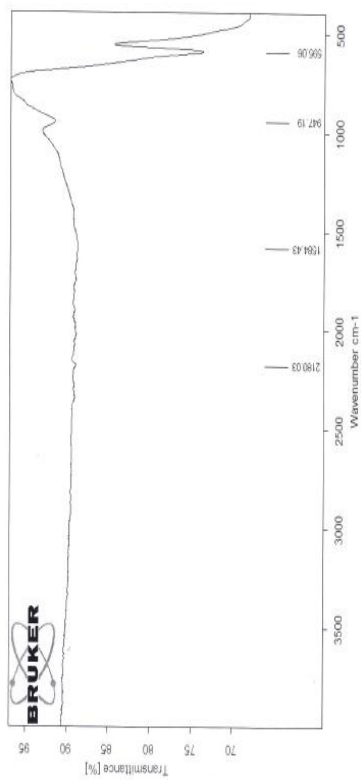


Fig. 3B

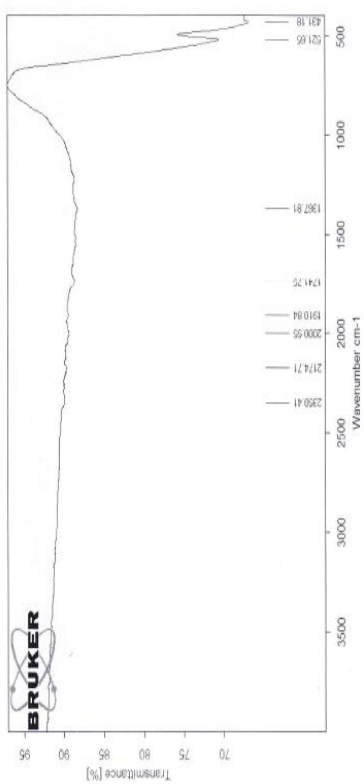


Fig. 3C

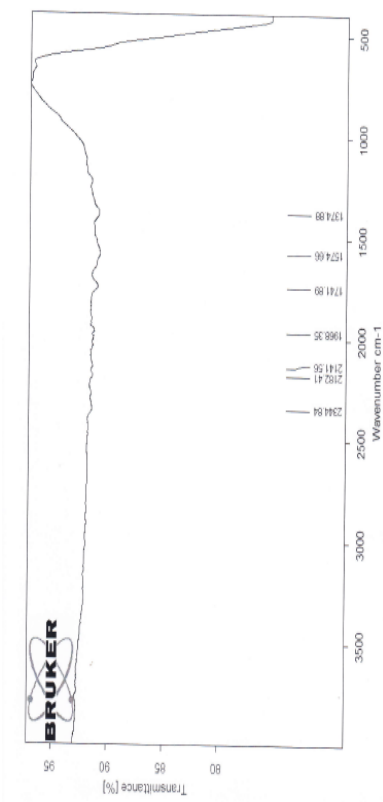


Fig. 3D

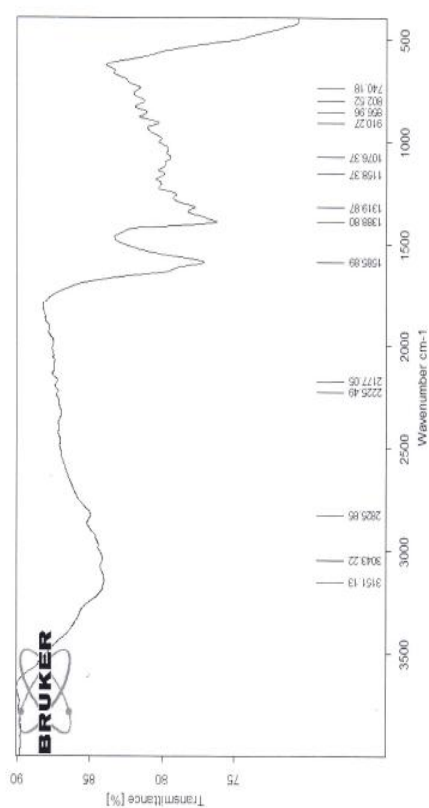


Fig. 3E

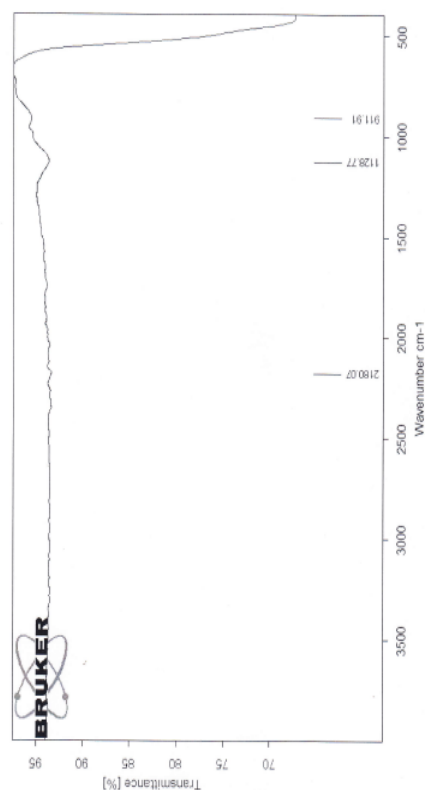


Fig. 3F

Fig. 3. FT-IR spectra of the metal oxides resulted from thermal decomposition of Gly complexes at 800 °C (A): Cr<sub>2</sub>O<sub>3</sub>, (B): MnO<sub>2</sub>, (C): Fe<sub>2</sub>O<sub>3</sub>, (D): NiO, (E): CuO and (F): ZnO.

### Acknowledgments

This Research was funded by The Deanship of Scientific Research at Princess Nourah Bint Abdulrahman University through the Fast-Track Research Funding Program.

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### تحضير أكاسيد $ZnO$ & $CuO$ & $NiO$ & $Fe_2O_3$ & $MnO$ & $Cr_2O_3$ باستخدام متراكبات الجلايسين<sup>2</sup> الخاصة<sup>2</sup> بهم كسلائف<sup>3</sup> للتحلل الحراري.

يهدف هذا البحث إلى تصميم وتشبيد وتعريف الشكل الجزيئي لبعض المشتقات الجديدة للحامض الأميني الجلايسين. و تم اختيار الجلايسين باعتبارها أبسط حامض أميني معروف وكذلك لما له من فوائد هائلة في صحة الإنسان. و بتكوين متراكبات الجلايسين الحديثة و المختلفة وجد أنها تلعب دوراً مهماً في حياة الإنسان. لذا تم تشبيد متراكبات  $Zn(II)$  &  $Cu(II)$  &  $Ni(II)$  &  $Fe(III)$  &  $Mn(II)$  &  $Cr(III)$  مع الحمض الأميني الجلايسين (Gly). ولقد تم إثبات هذه المتراكبات المحضرة حديثاً بواسطة التحليل الطيفي بالأشعة تحت الحمراء (FT-IR). تم الحصول على أكاسيد فلزية من كل من متراكبات الجلايسين وذلك باستخدام التحلل الحراري عند 800 درجة مئوية. و لقد تم التأكد بواسطة دراسة ال FT-IR للمواد الصلبة الناتجة عن تكوينها أنها بالفعل قد تكونت في شكلها النقي المتوقع و المفترض سلفاً.