International Journal of Engineering and Technical Research (IJETR)
ISSN: 2321-0869 (O) 2454-4698 (P), Volume-13, Issue-1, January-June 2023

Synthesis of complexes of rare earth elements mixed with metallic nutrients being used as fertilizers on melons

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***Abstract*— Rare-earth fertilizers with lactate (LA) and glutamate (GA) mixed with the metallic nutrients (Cu, Ag, Zn, Mo) were successfully prepared and characterized. The formulas of the as-prepared complexes were $LnLA_3.3H_2O$ and $LnGA_3$ (Ln: rare earth elements). The mixed fertilizers were employed for the cultivation of the Japanese melon Taki. The results demonstrated a significant enhancement in the quality and productivity of the target plant. The growth time of melon was shortened by about 14% and the yield increased by 1.5 times whereas the ability to resist common diseases and fungi was remarkably improved as compared to conventional foliar fertilizers.**

***Index Terms*— Rare-earth fertilizer, lactate complex, glutamate complex, metallic nutrient, melon cultivation.**

I. INTRODUCTION

The rare earth metals include the elements of the lanthanide series with Sc and Y. In recent years, REMs have been widely used in various aspects such as high-technological industry, pollution treatment, or reinforcement materials for weathering resistance[1–3]. In addition, many studies have also employed rare earth fertilizers for enhancing the productivity of cultivation[4,5]. REM fertilizers, containing nitrate forms, chloride forms, and complex forms with amino acids, have widely been used in many countries.

Glutamic acid (GA), also known as α-amino glutaric acid, is a vital protein amino acid in both animals and plants. Glutamate plays a central role in the amino acid metabolism of living organisms[6]. Glutamate has two carbonyl groups located at two heads of molecular. When forming complexes with metals, a carbonyl group is bound with metal ions whereas another carbonyl group is also in a free state to bind with another metal ion in the environment. This possibly increases micromineral absorption.

Lactic acid (LA), an organic acid, involves adjacent hydroxyl and carboxyl groups. When forming complexes with metals, the oxygen atoms on both the hydroxy group and carboxyl groups possibly coordinate with the metals. LA also plays a crucial role in glycolysis, an essential cycle for the growth of every living individual [7].

Therefore, the supplementation of lactate and glutamate for plants via REM fertilizers is expected to increase both the quantity and quality of plants. Moreover, recently, many studies have shown that the supplementary metallic nutrients in the form of nanoparticles during cultivation also promoted and protected the growth of plants in different stages [8–10]. Thus, this work attempted to synthesize the complexes of rare-earth elements with glutamic and lactic acids, followed by mixing with several metallic nutrients to obtain the REM fertilizer. Later, obtained fertilizers were used on the melon in Vietnam to evaluate its efficiency.

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II. EXPERIMENTAL

A. Materials

All chemicals including rare-earth oxides (Ln_2O_3) (99.9%), lactic acid, glutamic acid, $AgNO_3$, $NaBH_4$, $Cu(NO_3)_2.3H_2O$, $Zn(NO_3)_2$, $(NH_4)_6Mo_7O_2.4H_2O$, HNO_3, DTPA, PVA were purchased from the Sigma Aldrich and used without any further purification.

B. Experimental

1) Preparation of Ln (III) complexes

Ln_2O_3 was dissolved in concentrated HNO_3 ($n_{Ln} : n_{HNO3} = 1:3$) to obtain $Ln(NO_3)_3$ before an excess amount of NH_4OH was added to the solution to completely precipitate the $Ln(NO_3)_3$. The precipitated $Ln(OH)_3$ was then filtered then washed with deionized water to a pH of 7.

In the next step, $Ln(OH)_3$ was added to a 15-L bath containing LA 4M solution ($n_{Ln} : n_{LA} = 1:3$). The bath was kept stirring for Ln (III) to completely form the complexes with LA ligands. The obtained complexes were in the form of $LnLA_3.nH_2O$. A similar procedure was applied for the preparation of $LnGA_3.mH_2O$ by the addition of $Ln(OH)_3$ to the as-prepared bath containing GA 3M solution ($n_{Ln} : n_{GA} = 1:3$).

2) Preparation of metallic nutrients

For the preparation of the silver-containing solution, 31.48 g of $AgNO_3$ and $NaBH_4$ (w/w = 1:3) mixture was added into an as-prepared bath containing 4.5 L of distilled water and gently stirred. The solution was kept stirred at 80 ^oC until obtaining a yellowish brown. The reaction time was also recorded. Additional distilled water was added to the bath till the volume of the solution reached 5 L. A similar procedure was applied for the preparation of

copper-containing solution by adding 94.53 g of $Cu(NO_3)_2.3H_2O$ and $NaBH_4$ mixture (w/w = 1:4) to the water bath and keeping stirring until acquiring a dark-brown solution.

ZnO was prepared by gel combustion method. 1 L of $Zn(NO_3)_2$ 1M solution was added to a 2-L beaker containing PVA dissolved in distilled water (n_{Zn} : n_{PVA} = 1:2) on a magnetic stirrer. The mixture was kept stirred at 80 °C for 3 h. The resulting gel was dried before being calcined at 650 °C for 2 h to obtain ZnO. A similar procedure was employed for the preparation of MoO_3 using $(NH_4)_6Mo_7O_2.4H_2O$ precursor.

3) Preparation of fertilizers

50 L of REM fertilizers were prepared by adding a corresponding volume of the as-prepared metallic-containing solutions of Zn, Mo, Cu, and Ag to the Ln(III) complex-containing solution. The final fertilizer products involve Zn, Mo, Cu, Ag, and Ln with concentrations of 1.35, 3.5, 0.5, 0.2, and 120 g. L^{-1}, respectively.

C. Characterization methods

The content of REM was determined by titration with DTPA in a pH buffer from 3.8 to 4.0. The percentages of other metals were determined by the inductively coupled plasma mass spectroscopy (ICP-MS) method in Agilent Technologies 7900 (Japan). Thermal behaviors of obtained products were characterized by Labys Evo 1600 equipment (SETARAM – France). The contents of C and N in the complexes were determined by the elemental analyzer of Analytik Jena (Germany). The functional groups of obtained complexes were characterized by infrared spectroscopy by Agilent Technologies (Japan) within the wavelength of 400 – 4000 cm^{-1}. The structures and crystalline phases of prepared metallic nutrients were analyzed by X-ray diffraction (XRD) using a D8 Advance diffractometer of Bruker (Germany) with copper Kα radiation.

III. Results and Discussion

A. Structural characterization of prepared Ln (III) complexes

The contents of elements in the complexes were analyzed and reported in Table. 1 to predict the formula of prepared complexes.

Table 1. Composition analysis of prepared complexes

Compounds	Content (%)			Formulas
	Ln	***C***	***H_2O***	
$LnLA_3.nH_2O$	30.17	23.28	11.13	$LnLA_3.3H_2O$
$LnGA_3.mH_2O$	23.96	31.03	0	$LnGA_3$

Thermal behaviors of prepared complexes were evaluated by TG/DTA methods (Fig. 1). Both the curves for $LnLA_3.3H_2O$ and $LnGA_3$ show weight loss when the temperature increases from room temperature to about 250 °C. This loss was attributed to the evaporation of water trapped in the complex. For $LnLA_3.3H_2O$ (Fig. 1A), from 250 – 500 °C, there existed a weight loss of 36.41 wt% corresponding with two exothermic peaks at 300 °C and 410 °C, which was ascribed to the partial decomposition of organic components. The weight loss of 15.27 wt% of the complexes continued to occur when the temperature rose to 700 °C corresponding with an exothermic peak at 532 °C due to the complete decomposition of organic components to obtain Ln_2O_3. After 700 °C, no significant change in mass suggested that the final product was Ln_2O_3. The total remaining weight of 37.37 wt% was approximate to the content of Ln_2O_3 in the proposed complex $LnLA_3.3H_2O$. For $LnGA_3$ (Fig. 1B), in the temperature range of 200 – 300 °C, a small weight loss of 9.26% corresponding with an endothermic peak at 278 °C was attributed to water evaporation. The decomposition of GA was observed when the temperature increased to 600 °C, corresponding with a weight loss of 62.41 wt% and two exothermic peaks at 450 °C and 500 °C. There was no change in mass when the temperature was higher than 500 °C, suggesting that the final product was also Ln_2O_3. The total remaining weight of 28.23 wt% was equivalent to the estimated proportion of Ln_2O_3 in the proposed complex $LnGA_3$.

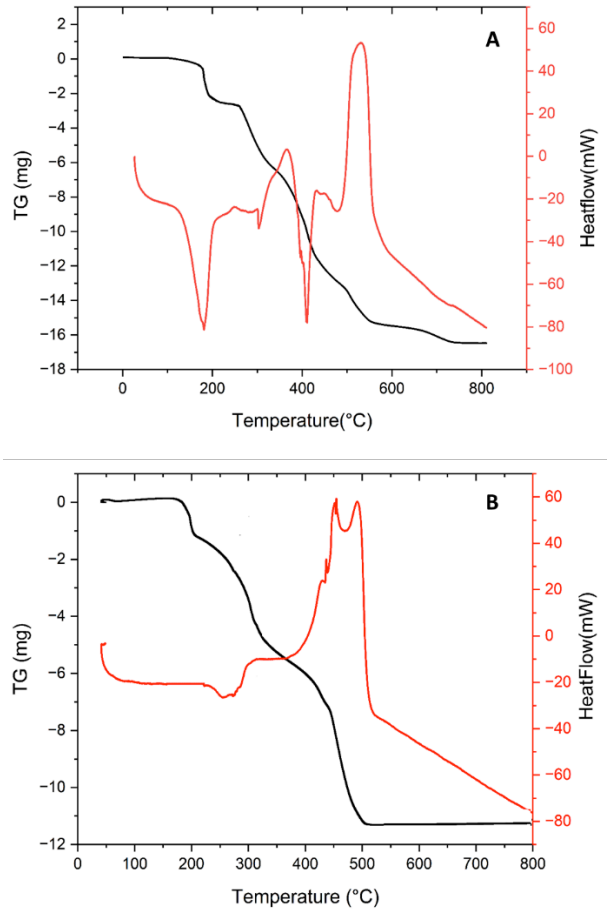

Figure 1. Thermal analyses of prepared complexes - A. $LnLA_3.3H_2O$, B. $LnGA_3$

To investigate the linkage and functional groups in the complexes further, FT-IR spectroscopy was employed. For IR spectra of $LnLA_3.3H_2O$ (Fig. 2A), a broadened peak in the range of 3000 – 3700 cm^{-1} was attributed to O-H stretching of water molecules in the complex. The weak signal of the $LnLA_3.3H_2O$ complex in the range of 2800 – 3000 cm^{-1} was ascribed to the stretching vibration of the $-CH_3$ group of LA. A spiked peak at 1552 cm^{-1} was specified for the carboxyl group of LA ligands which was a significant shift as compared to that in a free LA molecule. This shift suggested the formation of -COO-Ln- linkage instead of -COOH, which shifted the absorption to smaller wavelengths. Thus, the linkage of the complex can be proposed as the inset figure. For the IR spectra of $LnGA_3$

(Fig. 2B), no peak for the O-H stretching of water in the complex was observed. The absorption bands at 2600 – 3150 cm^{-1} and 2000 – 2200 cm^{-1} were characterized for amine groups of GA ligands. These absorption bands shifted to a lower wavelength as compared to the corresponding bands of free GA molecules. This was due to the bond formed between the -NH_2 group and Ln (III) ion. A sharp peak was also observed at 1635 cm^{-1} ascribing for the carboxyl group of GA. However, this peak was negatively shifted to a lower wavelength as compared to two carboxyl groups of a free GA molecule, suggesting the formation -COO-Ln- linkage between Ln (III) and one carboxyl group of GA ligand in the complex. A proposed structure of the $LnGA_3$ complex was described in the inset figure.

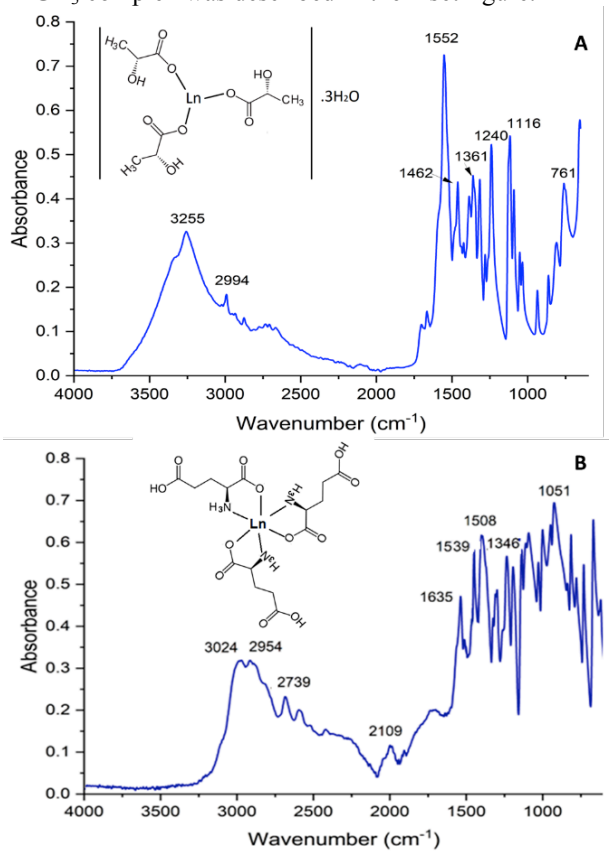

Figure 2. IR spectra of prepared complexes - A. $LnLA_3.3H_2O$, B. $LnGA_3$.

B. Structural analyses of prepared metallic nutrients

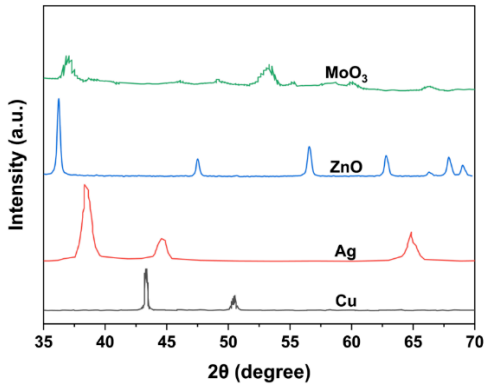

Figure 3. XRD patterns of prepared metallic nutrients

The structure and crystalline phase of prepared Cu, Ag, ZnO, and MoO_3 nutrients were examined by XRD analyses and are illustrated in Fig. 3. The characteristic peaks of Cu at 43.3° and 50.5° were exclusively assigned to the (111) and (200) planes, respectively. Meanwhile, the diffraction peaks of Ag were located at 38.3°, 44.6°, and 64.8°, which corresponded to the (111), (200), and (220) planes, respectively. ZnO formed by the gel combustion method had the specific diffraction at 36.2°, 47.5°, 56.6°, 62.8°, 67.9°, and 69.5° regard to the (101), (101), (110), (103), (200), and (112) planes, respectively, of the wurtzite structure. The diffraction patterns of MoO_3 were characterized at 37.1° and 53.3°, which corresponded to the (060) and (211) planes, respectively. These results were in agreement with previous studies about the corresponding metals[11–14].

C. Evaluation of Ln (III) complex fertilizer on the cultivation of Japanese melon Taki in Vietnam.

The evaluation of prepared REM fertilizer was conducted in an area of 360 m^2 divided into 5 segments. Four segments were tested with REM fertilizer and the rest employed the foliar fertilizer in the market under the label Thien Nong and followed the manufacturer's instructions. The REM fertilizer was tested for the capability of promoting the growth and yield of Japanese melon Taki. The spraying time and the dosage of REM fertilizer were used as follows.

- First time: melons were planted for 10 days with the ratio of REM fertilizer/water = 1/1000; the final content of REM fertilizer was 50 mg. L^{-1}.
- Second time: 15 – 20 mL of REM fertilizer was mixed in 16 – 20 L of water to spray for an area of 360 m^2.

The experiment results were reported in Table 2 and the growth of melons was captured in Fig. 4. The results demonstrate the significant enhancement of REM fertilizer for the growth of melons. The harvest time was shortened by 9 days whereas the total yields exhibit a 1.5-time increase from 36 tons/ha to 50 tons/ha. In addition, other indices for melons using REM fertilizer involving dissolved sugar content, the average weight of melons, the color of leaves, or the presence of harmful fungi and insects were also remarkably improved as compared to melons using no REM fertilizers.

Table 2. Comparison of Japanese melon Taki with and without using REM fertilizers

Criteria	Unit	Without REM fertilizers	With REM fertilizer
Time to down seed	dd/mm/yyyy	05/10/2022	
Time to make the bed	dd/mm/yyyy	15/10/2022	
Time to harvest	days	67	58
Number of melons per tree	melons	2	2
Average weight	g	1300 – 1800	1600 – 3000
Productivity	tons/ha	36	50
Sugar content	Brix	13 – 15	14 – 17
Color of leaves	-	Dark green, yellow	Dark green
Fungus	-	Yes	None
Leaf spots	-	Yes	None

Figure 4. The growth and development of melons sprayed with REM fertilizers – A. Downing seed, B. Making bed, C and D. Growing, E. Final product.

IV. Conclusion

REM fertilizers based on Ln (III) complexes mixed with nano metallic nutrients (Cu, Ag, Zn, Mo) were successfully prepared, characterized, and practically used on melon cultivations. The prepared fertilizers significantly reduced the harvest time of Japanese melon Taki from 67 days to 58 days while increasing the yield by about 50% as compared to conventional cultivation methods. The enhancements in average weight, sugar content, and ability to resist diseases of Japanese melon Taki using REM fertilizers have also prevailed

Acknowledgment

This research is funded by the Vietnam Academy of Science and Technology under grant number UDSXTN.05/22-23.

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