Spectroscopic (UV-Vis), Morphological (SEM-EDS) and Thermal (TG-DTG-DTA) Evaluation of Nickel Salts (Acetate, Chloride and Nitrate).

<u>Tamara Maria de Andrade¹*</u> (PG), Marins Danczuk¹ (PQ), Mariane Dalpasquale¹ (PG), Fauze Jacó Anaissi¹ (PQ)

¹Universidade Estadual do Centro-Oeste do Paraná, UNICENTRO-PR, Chemistry Department, Laboratory of Materials and inorganic compounds, LabMat/UNICENTRO, CEP. 85040-080, Guarapuava-PR, Brazil. * andrade.tamaraa@gmail.com

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Understand the general behavior of the precursors of an inorganic synthesis is extremely important, because it will influence directly in the synthesized product. In this work is presented a comparative and systematic study of different nickel salts (acetate, chloride and nitrate) from data of electronic spectroscopy (UV-Vis), thermal analysis (TG-DTG-DTA) and scanning electron microscopy (SEM-EDS).

Results and Discussion

The electronic spectra of nickel salts in solution have the same absorption behavior for the transitions (dd): ${}^{3}A_{2g} \rightarrow {}^{3}T_{1g}$ (P) in 394 nm, ${}^{3}A_{2g} \rightarrow {}^{3}T_{1g}$ (F), in 735 nm, ${}^{3}A_{2g} \rightarrow {}^{1}T_{2g}$, in 500 e ${}^{3}A_{2g} \rightarrow {}^{1}E_{g}$, in 660 nm. This is because, in aqueous solution, Ni²⁺ salts, without the presence of complexing agentes is the fomation of the green ion $[Ni(H_2O)_6]^{2+}$. Assigned to the transitions was calculed parameters Racah (B = 903,67 cm⁻¹) and 10 Dq (8133,00 cm⁻¹), consistent with the literature¹.

In Figure 1A it is observed that reflectance behavior of their salts and implications significant in the behavior of semiconductor salts (Figure 1B), in other words the values of the energy *band-gap* (E_{BG}).

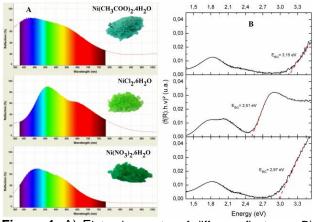


Figure 1. A) Eletronic spectra of diffuse reflectance. B) Corresponding energy spectra by Wood-Tauc method.²

The **Table 1** shows the experimental data obtained from thermal analysis curves (TG-DTG-DTA) of nickel salts. The hydration, except for nitrate were consistent to that indicated by the supplier. Unlike other salts, chloride showed a higher decomposition temperature and higher residue formation (NiO).

Morphological analysis (SEM-EDS) held TG residue confirms the formation of nickel oxide (NiO) as the final product for salts, which can be related to the nature of the cations (acetate, chloride and nitrate) and not measurable by EDS, or due to the thermal environment (heating ramp, gas flow rate of carrier, pan type). We highlight that the residue of nickel nitrate (NiO_{1.1}) presented the closest composition of the theoretical stoichiometry (NiO).

Table 1. Experimental data by TG-TG-DTA.

Nickel salt	H₂O (mols)	Residue (%)	DTA (°C)
Acetate	4,00	29,9	118 En.
[Ni(CH ₃ COO) ₂ •4H ₂ O]			378 Ex.
Chloride [NiCl ₂ •4H ₂ O]	6,03	31,1	206 En.
			237 En.
			705 En.
Nitrate	6,89	23,9	235 En.
[Ni(NO ₃) ₂ •6H ₂ O]			316 En.

Obs: En. Endothemic; Ex. Exothermic.

Conclusions

Inorganic salts evaluation becomes important from an analytical point of view, spectroscopic, structural, thermal and morphological, for understanding of characterization techniques and purposes of comparison with the derivatives prepared from these, as oxides, hydroxides and oxy-hydroxides.

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