Summary

Chapter I, Introduction

This chapter includes the classification of ceramic pigments, literature survey of the color ceramic pigment and its properties. This chapter explains the different methods of preparation of ceramic pigment as emulsion and co-precipitation method, hydrothermal method, sol-gel method, penchini method and low temperature combustion synthesis method.

Chapter II, Experimental

This chapter includes the description of the materials reagents and the preparation of different solution systems for cobalt and nickel salts. This part explains different fuels as Urea (U), oxalyl dihydrazide (ODH), 3-methyl-pyrazole-5-one (3MP5O) and N, N-bis-(3-amino-propyl) oxalamide (3APOA). It shows also the preparation of ceramic pigment powder for different $Co_xMg_{1-x}Al_2O_4$ and $Ni_yMg_{1-y}Al_2O_4$ systems by using different fuels.

This part also describe the instruments used such as Thermogravimetric, differential Thermal and differential thermogravimetric techniques (TG-DTA), X-ray diffraction techniques (XRD), Transmission electron microscopy (TEM), Fourier Transform Infrared Spectra (FT-IR), Diffuse reflectance spectra (DRS), UV- visible spectra and other methods that are using in analysis of ceramic pigment.

Chapter III, Result and discussion

First: The spectral characterization of $Co_xMg_{1-x}Al_2O_4$ systems ceramic pigment using different fuels.

Fourier Transform Infrared Spectra (FT-IR) of the powder pigment:

The figures of FT-IR spectra of different cobalt systems are present. All samples show the presence of bands for organic derivatives in low calcination temperatures. But, these function groups are disappeared with calcinations temperatures. The spectra exhibit that the normal $MgAl_2O_4$ and $CoAl_2O_4$ spinels formed up to 700^oC.

Diffuse reflectance spectra (DRS) of the powder pigment:

The diffuse reflectance spectra for cobalt systems using different fuels are presented. All samples show the appearance of blue color. The intensity of blue color is increased with calcination temperatures and doping percentage of cobalt cations. The blue color was measured by the CIE-L*a*b* colormetric method.

Electronic spectra of the powder pigment:

The visible spectra for cobalt systems with different doping percentage using different fuels are studied. All systems give trip-bard band in range 500-650 nm that characterized for cobalt electron transition to produce blue color.

Second: The spectral characterization of $Ni_yMg_{1-y}Al_2O_4$ systems ceramic pigment using different fuels.

Fourier Transform Infrared Spectra (FT-IR) of the powder pigment:

The figures of FT-IR spectra of different nickel systems are obtained. All samples show the presence of bands for organic derivatives in low calcination temperatures and function groups were disappeared with increasing the calcination temperatures. The inverse $MgAl_2O_4$ and $NiAl_2O_4$ spinels are formed up to 800^{o}C.

Diffuse reflectance spectra (DRS) of the powder pigment:

The diffuse reflectance spectra for nickel systems using different fuels are presented. All samples show the appearance of cyan (green-blue) color. The intensity of cyan color is increased with calcination temperatures and doping percentage of nickel cation. The cyan color was measured by the CIE- L*a*b* colormetric method.

Electronic spectra of the powder pigment:

The visible spectra for nickel systems with different doping percentage and using different fuels are studied. All systems give trip-bard band in range 500-650 nm that characterized for nickel electron transition to produce cyan color.

Third: Crystal structure characterization of $Co_xMg_{1-x}Al_2O_4$ systems ceramic pigments using different fuels.

Thermal analysis (TG-DTA) of the powder pigment:

The thermogravimetric analysis is studied and the decomposition of samples from room to1000 ^{o}C in steps is obtained and curves explain that all samples become more stable up to 700 ^{o}C. The phase under study is formed at this temperature. Differential thermal analysis are studied both the exothermic and endothermic reactions. All samples show the present of endothermic reaction for evolution of water molecules and phase formation. These samples show also the presence of exothermic peak as result of decomposition of organic material.

X-ray diffraction techniques (XRD) of the powder pigment:

The X-ray diffraction of cobalt systems are shown only small crystallites samples that indicated by the broadening lines at low calcined temperature. The calcined powders at different firing temperatures lead to the appearance of the crystalline sample. The average crystallite sizes are calculated from the X-ray diffraction peaks by using scherrer equation ($D=0.9\lambda/\beta\cos\theta$), where λ is wavelength, θ is the diffraction angle and β is the corrected halfwidth. The particle sizes in the range of nanosize and increase with the calcinations temperature. Also, particles size decrease as result of the increasing the percentage of cobalt doping. Density of sample can be calculated and compared with experimental values.

Transmission electron microscopy (TEM) of the powder pigment:

TEM photographs of cobalt samples show the sheet and spherical shapes depending on the percentage of cobalt ion doping. As a result of the increasing of amount of cobalt doping, the shape becomes more spherical and particle sizes are decreased.

Fourth: Crystal structure characterization of $Ni_yMg_{1-y}Al_2O_4$ systems ceramic pigments using different fuels.

Thermal analysis (TG-DTA) of the powder pigment:

The decomposition of samples are studied from room to1000^{o}C using thermogravimetric analysis and curves explain that all samples become more stable up to 700^{o}C. Differential thermal analysis studies the exothermic and endothermic reactions. All samples show the present of endothermic reaction for evolution of water molecules and phase formation. These samples show the present of exothermic peak as result of decomposition of organic material

X-ray diffraction techniques (XRD) of the powder pigment:

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Transmission electron microscopy (TEM) of the powder pigment:

TEM photographs of nickel samples show the sheet and spherical shapes depending on the percentage of nickel doping. As a result of the increasing of amount of nickel, the shape becomes more spherical and particle sizes decrease.

Chapter IV, Application of nano ceramic pigment on glaze

First: The preparation of colored blue glaze using $Co_xMg_{1-x}Al_2O_4$ systems of different fuels ceramic pigment powder.

The colored glaze prepared using 10% (wt/wt) of ceramic pigment powder to glaze using different systems of powder at 1100-1150^oC at different calcinations times for each system. The produced colored glaze characterized using diffuse reflectance spectra. The colored blue glaze is more intensity than the powder and the calcinated colored glaze for 30 minute is more than 15 minute.

Second: The preparation of colored green-yellow glaze using $Ni_yMg_{1-y}Al_2O_4$ systems of different fuels ceramic pigment powder.

The colored glaze prepared using 10% (wt/wt) of ceramic pigment powder to glaze using different systems of powder at 1100-1150^oC at different calcinations times for each system. The produced colored glaze characterized using diffuse reflectance spectra. The colored green-yellow (lime green) glaze is different than the cyan intensity for powder and the calcinated colored glaze for 30 minute is more than 15 minute in the same direction.

Third: The effect of acids and base on colored glaze using $Co_xMg_{1-x}Al_2O_4$, $Ni_yMg_{1-y}Al_2O_4$ systems and ceramic pigment powder.

The concentrated and diluted mineral acids such as sulphuric, hydrochloric, nitric acids are not effect on the pigment powder and colored glaze. But, hydrogen fluoride acid is effect on pigment after 14 days. Pigment and colored glaze are not effect by sodium and ammonium hydroxide.