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Mineralogical, Thermal and Morphological Analysis of some Clay Materials from Tunisia

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ABSTRACT

A combination of analytical techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), differential thermal analysis (DTA) and thermogravimetric analysis (TGA) were employed to characterize clays from different regions from Tunisia. The X-ray diffraction studies showed that the clay samples consist predominantly of clay minerals such as kaolinite, smectite, illite and halloysite, and quartz with trace amounts of magnetite, dolomite, calcite and gypsum minerals. The thermal analysis revealed thermograms that provided valuable information on the purity of materials and the mode of the reactions of the various clay samples.

Key words: Tunisian clays, energy dispersive analysis of X-ray, differential thermal and thermogravimetric analysis, scanning electron microscopy.

I. INTRODUCTION

Clays are raw materials abundantly found, and widely used by the ancient civilizations to make figures and ceramic artifacts (F. H. Norton, 1974, R. A. Habe et al, 1991). Nowadays, they are still used in the manufacture of ceramic products such as bricks, roofing tiles, porcelain, sanitary wares, wall tiles and floor tiles, and also are used in different industrial chemical processes. Clays are polymineralic fine-grained inorganic materials (< 2 µm), being constituted essentially of clay minerals and non clay materials as impurities. Clays are seldom pure. Each of these constituents contributes to the plastic forming and fired characteristics of the ceramic bodies. The clay minerals are hydrated aluminosilicates with layer structures that are responsible for the characteristic properties of the clays. There are a variety of clay minerals, including

kaolinite, halloysite, illite, montmorillonite, chlorite, among others (C. F. Gomes, 1988; P. S. Santos, 1989).

Clays are of immersed geological, industrial and agricultural importance (Murray, 1963; Ekosse, 1994). The mineral assemblage of clays helps in understanding and management of erosion and flood related problems (Kotoky *et al*., 2006), and in the construction of tunnels, road cuts, fills and dams (Oden *et al*., 2001). Depending on the physical and chemical characteristics, clays may find application in a number of industries such as plastics, paint, ceramics, ink, catalysts, pharmaceutical and fibre glass among others (Worall, 1975; Murray, 1980; Emufu rieta *et al*., 1992).

Thermal analysis involves a dynamic phenomenological approach to the study of materials by observing the response of these materials to a change in temperature. This approach differs fundamentally from static methods of analysis, such as structural or chemical analyses, which rely on direct observations of a basic property of material (*e.g.* crystal structure or chemical composition), at a well-defined set of conditions (*e.g.* temperature, pressure, humidity). Clay minerals are highly susceptible to significant compositional changes in response to subtle changes in conditions. For example, changes in the fugacity of water affect the stability of interlayer H2O in a clay mineral. Therefore, care must be taken that all experimental conditions are known with accuracy and precision. (S. Guggenheim et al, 2001).

In this paper the mineralogical composition of representative clay samples from different regions were examined. A wide range of techniques was employed, including X-ray diffraction (XRD), differential thermal analysis (DTA), thermogravimetry (TGA), scanning electron microscopy (SEM).

II. MATERIALS

In this work for clay samples from different regions of Tunisia: kairouan, tejra, gafsa and kasserine were used. The samples were labeled E1, E2, E3 and E4 respectively.

After crushing, the samples were subjected to various treatments to remove the clay fraction (M. Robert et al, 1974):

(1) Removal of rude material by sieving.

(2) Treatment with acid to the clay-limestone materials.

(3) Oxidant treatment for the removal of organic matter.

(4)Wash with distilled water to obtain a suspension.

(5) Extraction of less than 2 µ m adopted by regulation under the law of particles stocks.

(6) Drying at 60 ° C.

(7) Spraying.

X-ray diffraction analysis was performed on clay simples' powder. TGA /DTA and SEM were performed on fine fraction less than 2 µ m.

III. METHODS

X-ray diffraction analysis was performed (URD-65 Diffractometer, Seifert) using monochromatic Cu-kα radiation at 40 kV and 40 mA over non-oriented specimens. Scanning speed was 1.5º (2θ)/min. The phases were identified from peak positions and intensities using reference data from the JCPDS handbook.

The morphology and texture of the clay particles were determined by scanning electron microscopy, using a Zeiss DSM 962 SEM coupled with EDS (energy dispersive spectroscopy).

TGA /DTA were carried out on the as-received samples (SDT - 2960 Simultaneous TGA-DTA, Instruments) under air atmosphere from room temperature up to 1150 ºC at a heating rate of 10 ºC/min.

IV. RESULTS AND DISCUSSION

X-ray diffraction of the clay samples is shown in Figs. 1-4. The sample clays have different mineralogical composition. The mineralogical phases found in E1, E2, E3 and E4 are shown in table1.

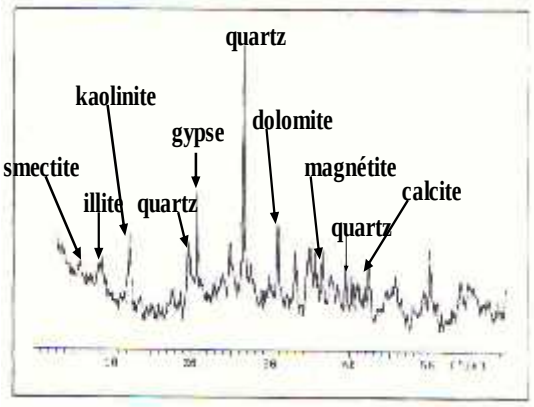


Figure 1: X-ray diffraction pattern of the sample clay E1.

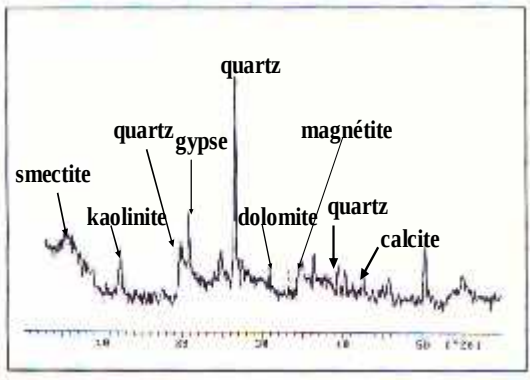


Figure 2: X-ray diffraction pattern of the sample clay E2.

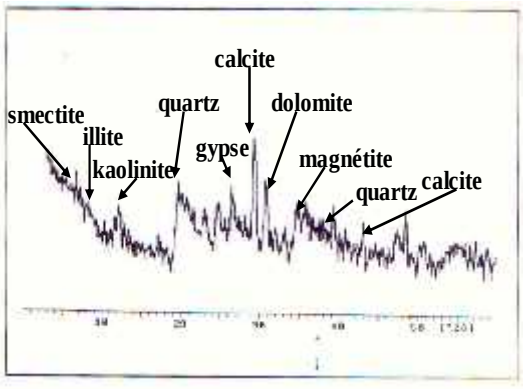


Figure 3: X-ray diffraction pattern of the sample clay E3.

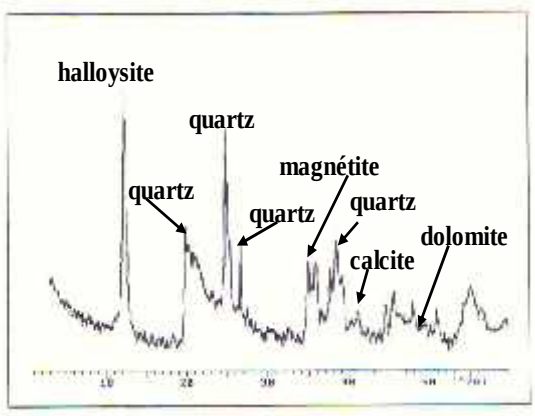


Figure 4: X-ray diffraction pattern of the sample clay E4

.Table 1. Mineralogical composition of the clay samples.

Clay samples	mineralogical composition
E1	kaolinite, smectite, quartz gypsum,dolomite, magnetit and calcite
E2	kaolinite, smectite, quartz gypsum,dolomite, magnetit and calcite
E3	kaolinite, smectite, illite quartz gypsum,dolomite, magnetit and calcite
E4	Halloysite, quartz gypsum,dolomite, magnetit and calcite

Morphological aspects of a clay sample obtained by SEM are shown in Figs.5-8. Kaolinite, smectite and illite plates with size below 2 µm, leading to pile up to agglomerates were observed.

In addition, it was also observed tubular particles typical of halloysite in figure 8. These results obtained by SEM are very interesting, because halloysite usually is not detected by other characterization techniques. In the case of the X-ray diffraction, the halloysite main peak is overlapped with the peak of kaolinite (*G. P. Souza et al*, 2005). Sometimes halloysite is also not detected if is present in a small amount.

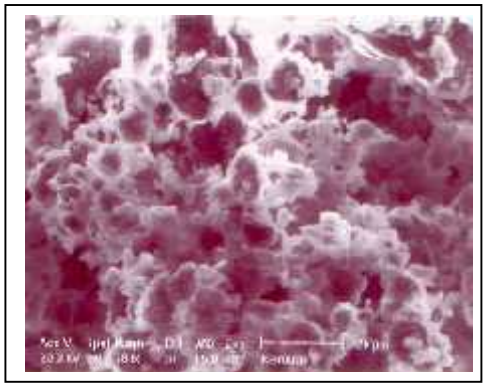


Fig.5 SEM observation of E_1

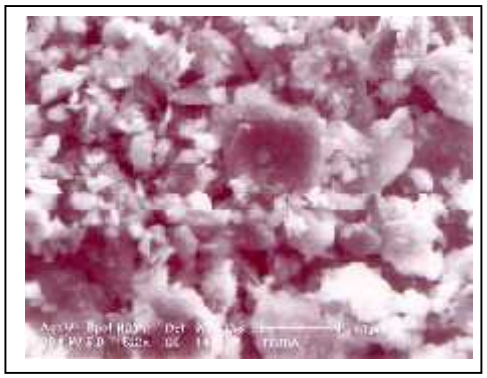


Fig.6 SEM observation of E_2

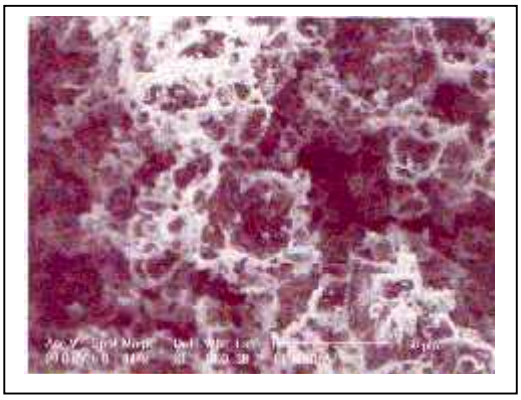


Fig.7 SEM observation of de E_3

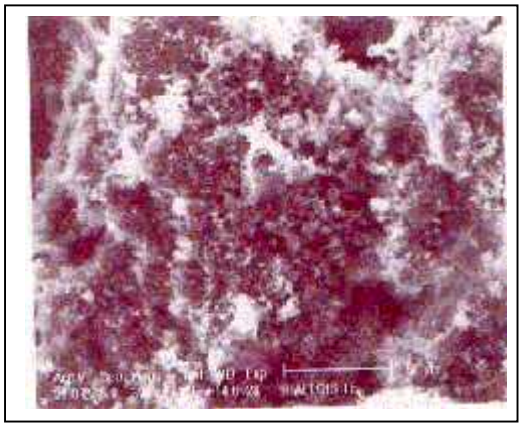


Fig.8 SEM observation of de E_4

The DTA curves show the effect of energy changes (endothermic or exothermic reactions) in a sample. For clays, endothermic reactions involve desorption of surface H2O (*e.g.* H2O on exterior surfaces) and dehydration (*e.g.* interlayer H2O) at low temperatures (<100°C), dehydration and dehydroxylation at more elevated temperatures, and, eventually, melting. Exothermic reactions are related to recrystallization at high temperatures that may be nearly concurrent with or after dehydroxylation and melting. Discriminating between desorption and dehydration or dehydration and dehydroxylation may be problematic. The TG curves ideally show only weight changes during heating. The derivative of the TG curve, the DTG curve, shows changes in the TG slope that may not be obvious from the TG curve. Thus, the DTG curve and the DTA curve may show strong similarities for those reactions that involve weight and enthalpy changes, such as desorption, dehydration and dehydroxylation reactions.

The results of thermo analytical analysis of clays are presented in Figures 9-16. The DTA thermograms showed 2 peaks, an endothermic and exothermic between 0° to 1000°C. The endothermic peaks which are asymmetric with a maximum at 535°C can be associated with the removal of last traces of OH in the form of H2O which can exist in the lattice even above 600°C. An exothermic peak around 320 ºC (E4) due the organic matter decomposition was observed for all studied samples. In addition, an exothermic event within the 890 - 900 ºC range was observed. This thermal event can be related to transformation of the metakaolinite to a spinel structure or a Si-containing γ-Al2O3 and amorphous silica In addition, it is also possible primary mullite be formed (G. P. Souza et al; 2005).

The TGA results of E5 reveal a single broad loss of mass in the region of 400 to 550°C. The total loss of 13.59% corresponds to the removal of water molecules in the kaolinite groups (V. N. Osabor et al, 2009) (calculated loss of mass of 10.76%). The dehydroxylation of clays occurs within this temperature range. No loss was observed above 550°C indicating a complete separation of the 2 weight loss steps on heating to a constant temperature of 500°C (Pekene and Sharp, 1974).

Two weight loss events are seen in the DTG curves of E1, E2, E3 and E4 at 54.10 - 56.56 ºC and 492.50 - 493.00 ºC, whose total weight loss is in the 11.59 - 12.60 % range. The first weight loss is related to the evolution of the physically adsorbed water by the kaolinite, smectite and illite particles. The second weight loss is associated to dehydroxylation of smectite, illite and kaolinite, which transforms into metakaolinite (G. P. Souza et al, 2005).

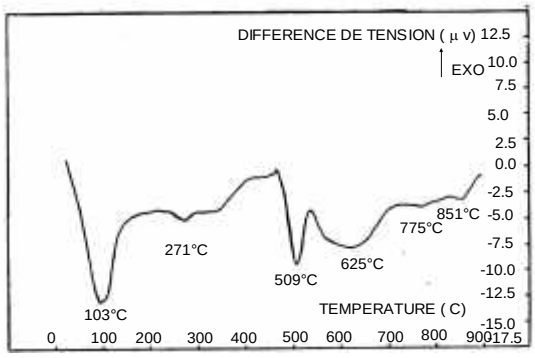


Fig.9 DTA curve of the clay sample E1.

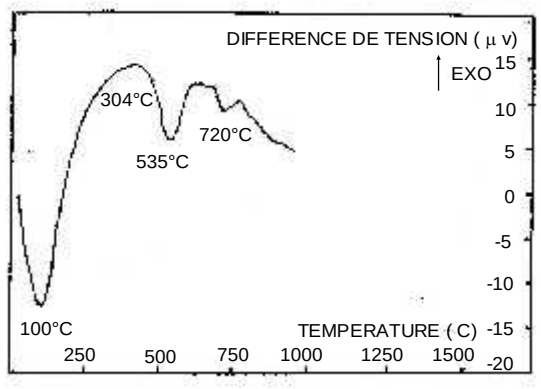


Fig.10 DTA curve of the clay sample E2.

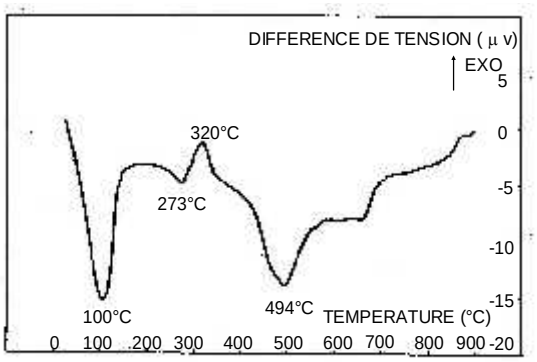


Fig.11 DTA curve of the clay sample E3.

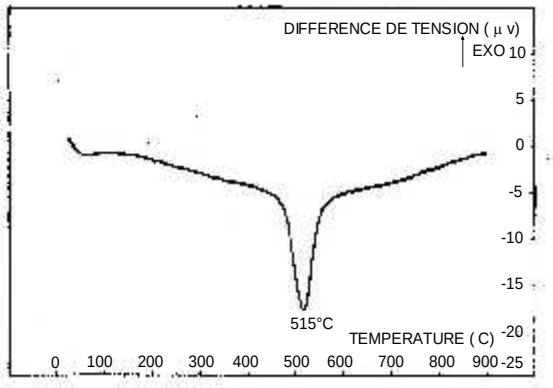


Fig.12 DTA curve of the clay sample E4.

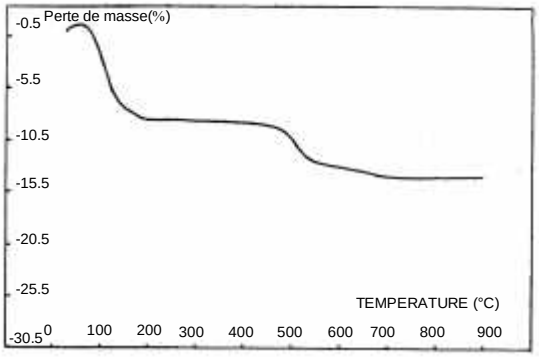


Fig.13- TGA curve of the clay sample E1

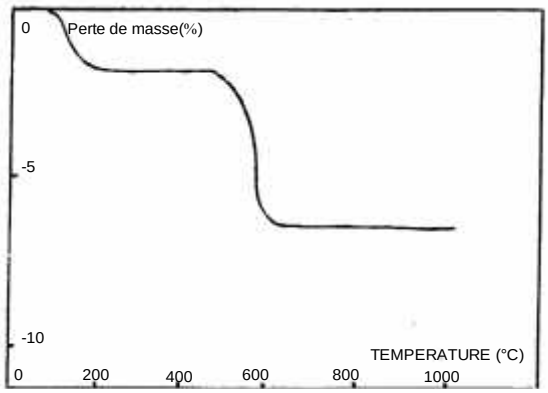


Fig.14- TGA curve of the clay sample E2

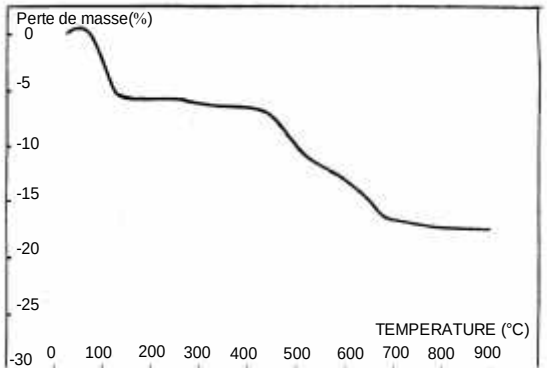


Fig.15- TGA curve of the clay sample E3

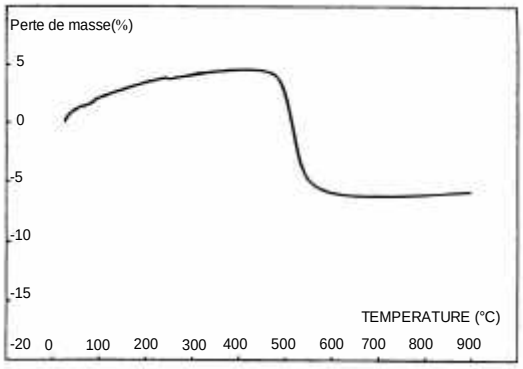


Fig.16- TGA curve of the clay sample E4

V. CONCLUSION

Clays from different regions from Tunisia have been characterized using XRD, SEM and DTA/TGA methods of analysis. From the results obtained, the following conclusions have been drawn:

(1) The clays have kaolinite, smectite, illite and halloysite as the minerals phase, and quartz, gypsum, calcite and magnetite as impurities.

(2) SEM analysis indicated the presence of tubular halloysite and Kaolinite, smectite and illite plates.

(3) The DTG/DTA curves suggest that the character of these clays is predominantly consisting of mineral clays.The sample of Ksserine (E5) is predominantly haloysite with minor contents of impurities. This sample is amorphous at 550°C.

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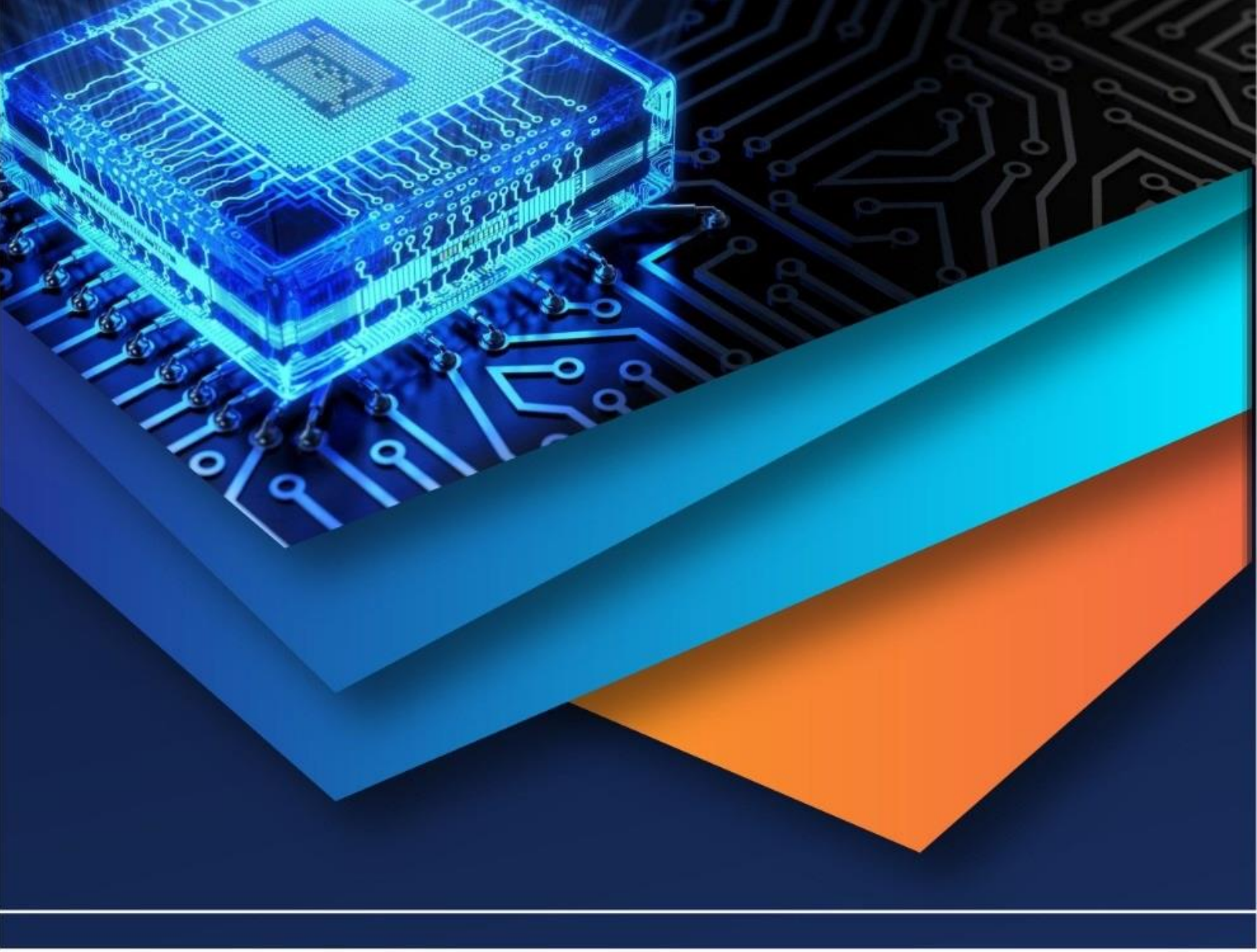
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