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Preparation and Characterization of ($Ba_{(0.99)}$ $Fe_{(0.01)}$ $Ti_{(0.99)}$ $Zr_{(0.01)}$ O_3) Nanostructure Perovskite using Energy Dispersive X-ray Microanalysis and Laser Induced Breakdown Spectroscopy Techniques

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Abstract
The objective of this study was to analyze of a single perovskite oxide sample with a chemical formula (Ba (0.99) Fe (0.01) Ti (0.99) Zr (0.01) O3) by using Energy Dispersive X-ray Microanalysis Spectroscopy (EDX) and Laser Induced breakdown Spectroscopy (LIBS). The sample was prepared through a solid-state reaction method at ($1200^{\circ}C$) and stored in 5ml plastic container for 60 days to verify the interaction between the powder and the plastic. The EDX revealed the presence of chemical elements such as (Barium, Titanium, Sulfur, Iron, Zirconium, Strontium, Thulium, Niobium, Zinc, Copper, and Osmium) which were found with different percentage values such as (73.976,24.801,0.537,0.183,0.176,0.163, 0.070,0.030,0.028,0.027,0.009 %) respectively. Also, this sample was irradiated by Nitrogen laser with pulse energy of (200 mJ), and the LIBS analysis showed that the neutral number of atoms for example (Fe, Ti, Os, Tm, Ba, Zr, Cu, O, Nb, and Zn) were appeared at this sample. In addition, the ions of many atoms for instance (Zr+3, Ti+3, Cu+3, Fe+5, Ti+6, Ba+3, S+2, Fe+2, O+2, S+5, Sr+2, Ba+2, and O+3) were presented at the end of this experimental work. The results obtained may be attributed to interaction between powders and plastic, and the difference in results is due to the accuracy of measurements for different techniques.

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Keywords
Perovskite Oxide, Interaction, Solid State Reaction Method, Ions, Storage Period

1. Introduction:

The nanostructure perovskite are materials that have a unique composition known as ABX_3, where x represents anions, such as oxygen or halogens; B typically stands for transition metal elements like cations of titanium; and A represents large metal cations, commonly alkaline earth or rare earth elements such as barium [1]. Among these materials, barium titanate ($BaTiO_3$) is a typical perovskite compound that is utilized in infrared sensors and electromechanical transducers due to its significant pyroelectric properties and large dielectric constant [2].

To utilize $BaTiO_3$ ceramics as a capacitor material, achieving a high dielectric constant and low loss factor, in addition to good density, is crucial. Barium titanate is a well-known ferroelectric crystalline material that possesses chemical mechanical stability and ferroelectric properties at and above room temperature, and can be easily prepared in the form of ceramic polycrystalline samples [3].

EDX technique is utilized to detect chemical metals and determine their concentrations in different environmental samples such as soil, powders, and liquids [4]. The technique utilizes the X-ray spectrum emitted by a solid sample that is bombarded with a focused beam of electrons to obtain a localized chemical analysis [5]. Qualitative analysis is relatively straightforward since it involves identifying the lines in the spectrum, which are simple to discern in X-ray spectra [6]. However, quantitative analysis, which involves determining the concentrations of the elements present, requires measuring line intensities for each element in the sample and comparing them to those in calibration standards of known composition [7]. By scanning the beam in a television-like raster and displaying the intensity of a selected X-ray line, element distribution images or 'maps' can be produced [8]. The scanning electron microscope EDX, which is closely related to the electron probe, is designed primarily to produce electron images, but can also be used for element mapping, and even point analysis, if an X-ray spectrometer is added. As a results, there is significance of instruments [9].

LIBS is an optical emission spectroscopy technique that is utilized to measure the concentrations of different elements in a material [10]. To generate a plasma plume (partially ionized gas) in the temperature range of 4,700- 19,700°C, a laser pulse is directed at the surface of the sample, ablates a small amount of material in the range of (1 ng), and is focused to a microscopic point on the sample [11]. Despite the low energy of the laser, the focused beam generates the plasma [12]. This plasma dissociated the matter comprising the sample into excited atoms and ions undergo transitions of electrons from lower to higher energy levels of the valence shell and, as they return to their ground state (transition from higher to lower level of valence shell), they emit characteristic lines from each element [13].

On the other hand, some perovskite oxides compounds have a unique nanostructure because the size of the atoms is on the nanoscale after preparation in experimental science physics laboratories [14]. In addition, most literature reviews in this field indicated that the grain sizes of these materials were measured in nanometers as shown below:

Nanosized powders and ceramics of Co-doped $BaTiO_3$ were investigated by Cui et al., (2021) via sol–gel process when they mixed all of material together. The characteristic of absorption frequencies found at the chemical bonds such as, O-H, C-O, Ba-O, and Ti-O. The XRD results showed that there is a cubic structure of $BaTiO_3$. The SEM micrographs of the free surfaces of ceramics confirmed that average grain size of these ceramics was 6.0 nm [15].

The nanostructure of BaTiO3 powder were proved by Emre et al., (2023) by using some spectroscopy techniques like XRD and SEM. The X-ray diffraction analysis confirmed that the samples had a cubic phase at room temperature. Also, the SEM results revealed that the morphologies and the particle sizes of the synthesized $BaTiO_3$ was 18.97 nm. [16].

The objective of this study was to utilize spectroscopy techniques such as EDX and LIBS to detect and measure the concentrations of chemical elements present in the nanostructure Perovskite ($Ba_{(0.99)}$ $Fe_{(0.01)}$ $Ti_{(0.99)}$ $Zr_{(0.01)}$ O_3) after it was stored in plastic container for (60 day) to verify the interaction between the powder and the plastic after the peroration process.

2.Material and Methods:

This part describes the experiment part including materials, equipment and methods, and spectroscopy techniques as shown below:

2.1. Raw materials:

The high purity raw powders (exceeding 99%) of Barium Carbonate ($BaCO_3$), Titanium dioxide (TiO_2), Magnesium oxide (FeO), and Manganese dioxide (ZrO_2) (The source should be mentioned) were used in this study.

2.2. Preparation of ($Ba_{(0.99)}$ $Fe_{(0.01)}$ $Ti_{(0.99)}$ $Zr_{(0.01)}$ O_3) Nanostructure Perovskite:

One nanostructure perovskite oxide sample which has a new chemical formula ($Ba_{(0.99)}$ $Fe_{(0.01)}$ $Ti_{(0.99)}$ $Zr_{(0.01)}$ O_3) was prepared by solid state reaction method. Initially, and 2gm of the powders were taken and mixed thoroughly with a small amount of acetone for 2hr to ensure homogeneity. Subsequently, the mixed powders were heated at 200˚C for 4hr in a laboratory oven after which it was sintered at 1200°C for 5 hours and the resulting solid sample was ground as shown in figure (1) below [17]:

Figure (1): CARBOLITE –CWF- Serial No: 20-302426, Manufacturing in England

The sample was stored in a plastic container with a volume of (5ml) which was manufactured from polyethylene material for a period of (60 days) to the possibility of interaction between powders and plastic made of polyethylene materials [18].

3. Results and Discussion:

The concentrations of the elements at ($Ba_{(0.99)}$ $Fe_{(0.01)}$ $Ti_{(0.99)}$ $Zr_{(0.01)}$ O_3) nanostructure perovskite were stored in a plastic container for (60) days, and they were investigated using EDX and LIBS spectroscopy techniques as shown below:

3.1. The Results of EDX Spectroscopy Technique:

Table (1): The (EDX) results of ($Ba_{(0.99)}$ $Fe_{(0.01)}$ $Ti_{(0.99)}$ $Zr_{(0.01)}$ O_3) nanostructure perovskite:

The Element	The Concentration of Element (%)
Ba	73.976
Ti	24.801
S	0.537
Fe	0.183
Zr	0.176
Sr	0.163
Tm	0.070
Nb	0.030
Zn	0.028
Cu	0.027
Os	0.009

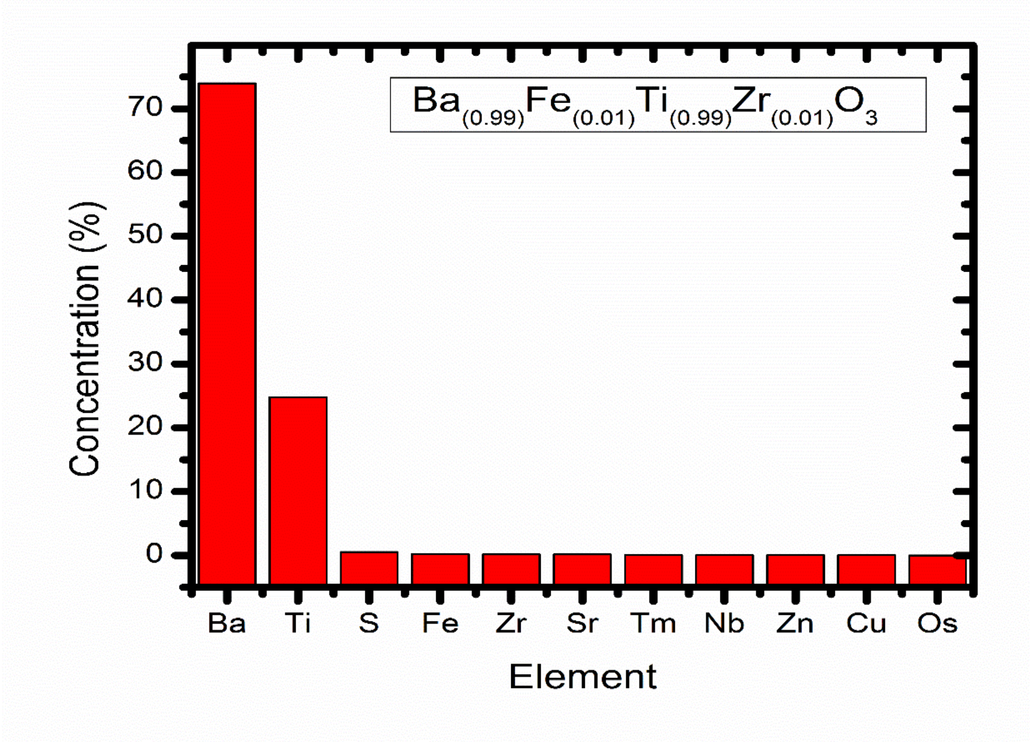

Figure (2): the EDX results (Ba $_{(0.99)}$ Fe $_{(0.01)}$ Ti $_{(0.99)}$ Zr $_{(0.01)}$ O_3) nanostructure perovskite

Table (1) and Figure (2) present the findings of the study after the prepared sample was stored for (60) days and analyzed using EDX spectroscopy technique [19]. The results confirmed that the presence of atoms of (Ba and Ti) of percentages of (73.976 and 24.80) respectively, which may be related to the normal structure of barium titanate compound [20]. Additionally, the results using this technique indicated that some other chemical elements such as (Fe and Zr) were found in the EDX spectrum of this sample with varying concentrations of (0.537 and 0.183) successively, which may be attributed to the doping process of the barium titanate compound with iron and zirconium [21]. After storage period, seven other metals for instance (S, Sr, Tm, Nb, Zn, Cu, and Os) appeared with concentrations of (0.176, 0.163, 0.070, 0.030, 0.028, 0.027, and 0.009 %) consecutively [22]. These results may be associated with the interaction between the powders and plastic container which was made from polyethylene material, and the migration of atoms during this chemical process after storage period.

In fact, the polythene plastic used for many uses; for example, storing powders under specific conditions during different time periods, and many changes that can occur in the composition of the powders [23]. During the long storage period, there is an interaction that may happen between powder atoms polyethylene material, and this process includes migration atoms of some elements of powder that combines with plastic atoms [24]. These potential toxic metals are highly persistent, and can bio accumulate in the tissues of biological organisms, resulting in severe health issues in the process over time [25].

3.2. The Results of LIBS Spectroscopy Technique:

Figure (3) and Table (2) verify the presence of several atoms in the newly prepared powder sample after it was stored for (60 days). These atoms were detected in varying concentrations, as outlined:

Table (2): The LIBS results of ($Ba_{(0.99)}$ $Fe_{(0.01)}$ $Ti_{(0.99)}$ $Zr_{(0.01)}$ O_3) nanostructure perovskite:

Wavelength (nm)	Intensity	Element
385.736503	129.730128	Zr III
432.192846	129.671128	Ti III
443.712508	127.829008	Nb I
446.167518	127.829008	Fe I
458.631415	127.593007	Cu II
483.559209	128.68779	Ti I
511.886248	127.652008	Fe V
523.40591	128.68779	Ti XXI
525.105532	127.711008	Ti I
548.333704	127.711008	Nb I
576.471895	128.74679	Os I
593.090424	127.770008	Ti II
613.108198	128.635346	Ba III
614.618974	133.702813	Ti I
616.318596	132.667031	Tm I
628.027105	127.652008	Fe I
651.255277	127.711008	Ti I
652.954899	127.016116	Cu II
669.384581	130.536465	Ba I
677.693846	127.652008	S II
719.995557	128.74679	Zr I
677.693846	127.711008	Fe II
686.00311	127.711008	Fe II
719.995557	128.635346	Ti II
740.768718	129.553128	O II
743.223728	129.553128	Ti I
754.17685	128.635346	Fe I
778.915796	128.635346	Fe II
782.315041	126.963671	Fe II
800.633193	129.612128	Cu I
810.453233	127.593007	Fe II
812.908243	127.711008	S V
823.861364	127.770008	Fe I
835.381026	127.540563	Cu II
850.299933	128.68779	Fe II
856.909576	128.68779	Ti I
868.618085	127.593007	Sr II
871.073095	127.593007	Ba II
881.837369	127.593007	Fe I
895.245501	127.481562	Fe II
911.86403	129.612128	O I
913.374806	127.711008	O III
915.829816	127.711008	Ba III
923.383693	128.68779	Ba III

928.293713	128.635346	Fe II
930.748723	128.635346	Ba I
948.311486	129.671128	O I
958.320373	127.770008	Fe II
959.831149	127.770008	Fe II
969.840036	127.711008	Ba III
989.857809	128.68779	Fe I
1001.37747	127.540563	Zn I
1013.08598	127.652008	Ba I
1021.39525	128.635346	Fe V
1022.90602	127.770008	Fe I
1036.31415	127.652008	Fe I
1047.83381	128.635346	Ti II
1049.53344	128.3469	Ti I

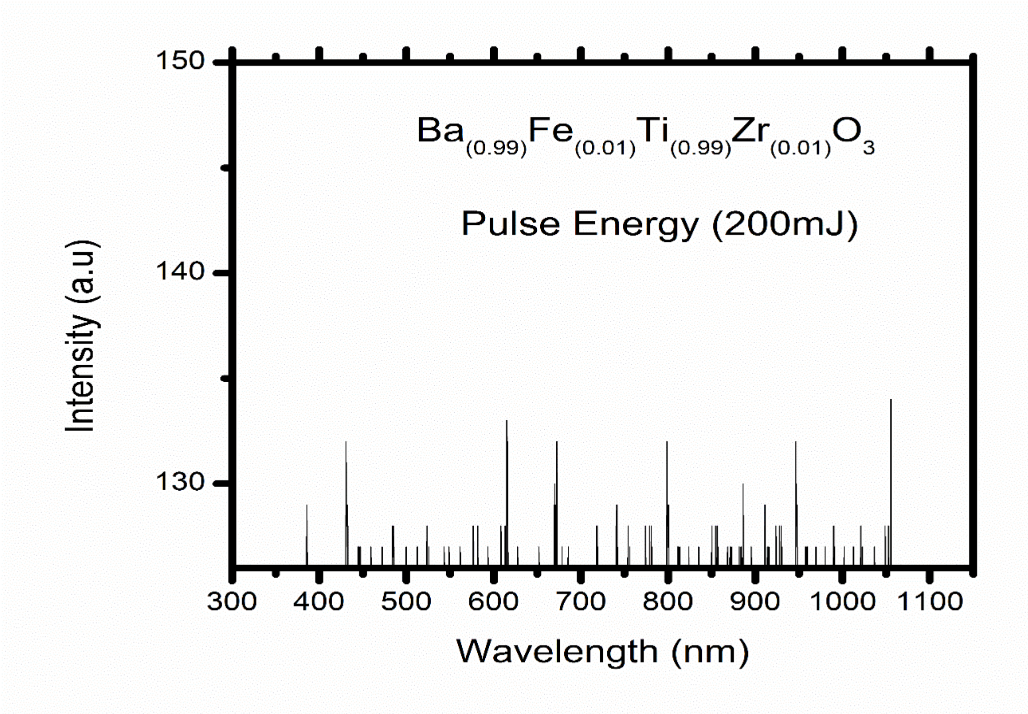

Figure (3): the LIBS results of (Ba $_{(0.99)}$ Fe $_{(0.01)}$ Ti $_{(0.99)}$ Zr $_{(0.01)}$ O_3) nanostructure perovskite

In this study, the samples were irradiated by a Nitrogen laser with pulse energy of 200 mJ, and the spectra analysis using LIBS technique revealed the detection of neutral atoms like (Fe, Ti, Os, Tm, Ba, Zr, Cu, O, Nb, and Zn) [26]. It is noteworthy that higher ionization states of the elements present in this sample, such as (Zr^{+3}, Ti^{+3}, Cu^{+3}, Fe^{+5}, Ti^{+6}, Ba^{+3}, S^{+2}, Fe^{+2}, O^{+3}, S^{+5}, Sr^{+2}, Ba^{+2}, O^{+3}) were also detected at the end of this experimental work when the oxygen ions are positive sign [27]. The results obtained from the analysis of Figure (3) and Table (2) after storing the prepared sample for 60 days using LIBS spectroscopy technique are attributed to the interaction between powder and plastic container and the migration of atoms during this chemical process [28]. However, powder is filled into transparent plastic bottles made from polyethylene terephthalate (PET) and stored for (60 days) days [29]. During this period there is an interaction between powder and plastic, and many elements can be found after this physical experiment such as (Sr, Tm, Ba, Ti, Zr, Os, and Zn) [30].

4. Conclusion:

This study investigated the impact of storing of ($Ba_{(0.99)}$ $Fe_{(0.01)}$ $Ti_{(0.99)}$ $Zr_{(0.01)}$ O_3) nanostructure perovskite in a 5ml plastic container for 60 days on its physicochemical properties. The concentrations of various atoms, including (Ba, Ti, S, Fe, Zr, Sr, Tl, Nb, Zn, Cu, Os, and O) were analyzed using EDX technique, which revealed percentage of (73.976, 24.801, 0.537, 0.183, 0.176, 0.163, 0.070,0.030,0.028,0.027,0.009%) respectively. The LIBS was also used, identifying the presence of atoms such as (Fe, Ti, Os, Tm, Ba, Zr, Cu, O, Nb, and Zn) in the sample. Additionally, several ions of these atoms, including (Zr^{+3}, Ti^{+3}, Cu^{+3}, Fe^{+5}, Ti^{+6}, Ba^{+3}, S^{+2}, Fe^{+2}, O^{+2}, S^{+5}, Sr^{+2}, Ba^{+3}, and O^{+3}) were present at the end of this experimental work. The results suggested an interaction between the powder molecules, which were manufactured from polyethylene material, and the effect of the storage period, which affects the physicochemical properties of the new nanostructure perovskite.

5. Recommendations:

1-Studying the optical properties of ($Ba_{(0.99)}$ $Fe_{(0.01)}$ $Ti_{(0.99)}$ $Zr_{(0.01)}$ O_3) nanostructure perovskite using UV-visible spectroscopy technique.
2- Studying the structural properties of (Ba (0.99) Fe (0.01) Ti (0.99) Zr (0.01) O3) nanostructure perovskite using X-ray diffraction spectroscopy technique.

6. Abbreviations:

Ba: Barium Atom.
Ba^{+3}, and Ba^{+3}: Barium Ions.
Cu: Copper Atom.
Cu^{+3}: Copper Ion.
EDX: Energy Dispersive X-ray Microanalysis Spectroscopy.
LIBS: induced breakdown Spectroscopy.
Fe: Iron Atom.
Fe^{+2}, and Fe^{+5}: Iron Ions.
Nb: Niobium Atom.
O^{+2}, and O^{+3}: Positive Oxygen Ion.
Os: Osmium Atom.
PE: Polyethylene material.
Sr: Strontium Atom.
Sr^{+2}: Strontium Ion.
S: Sulfur Atom.
S^{+2}, and S^{+5}: Sulfur Ions.
Tl: Thulium Atom.
Ti: Titanium Atom.
Ti^{+3} and Ti^{+6}: Titanium Ions.
Zn: Zinc Atom.
Zr: Zirconium Atom.
Zr^{+3}: Zirconium Ion.

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تحضير وتوصيف مادة البيروفسكايت النانوية التركيب $(Ba_{(0.99)}\ Fe_{(0.01)}\ Ti_{(0.99)}\ Zr_{(0.01)}\ O_3)$ بواسطة تقنيات التحليل الطيفي المجهري للأشعة السينية المشتتة للطاقة والانهيار المستحث بالليزر

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الملخص

يهدف هذا البحث إلى تحليل عينة أكسيد البيروفسكايت الأحادي ذات الصيغة الكيميائية (Ba (0.99) Fe (0.01) Ti (0.99) Zr (0.01) O3) وذلك بواسطة تقنية التحليل الطيفي المجهري للأشعة السينية المشتتة للطاقة (EDX) وتقنية الانهيار المستحث بالليزر(LIBS). هذه العينة تم تحضيرها بواسطة طريقة تفاعل الحالة الصلبة عند (1200) درجة مئوية وهذه العينة تم تخزينها لمدة (60 يوما) في حاوية بلاستيكية سعة (5مليلتر) وذلك للتأكد من مدى تفاعل المسحوق والبلاستيك. نتائج تقنية (EDX) أكدت وجود عدد من العناصر الكيميائية مثل (الباريوم، التيتانيوم، الكبريت، الحديد، الزركونيوم، الاسترنشيوم، الثليوم، النيوبيوم، الزنك، النحاس، والأوزميوم) عند النسب المئوية (73.976، 24.801، 0.537، 0.183، 0.176، 0.030، 0.070، 0.163، 0.028، 0.027، و0.009%) على التوالي. أيضاً، هذه العينة تم تعريضها لاحقاً إلى التشعيع بواسطة الليزر من نوع النيتروجين عند طاقة نبضة (200mJ)، ونتائج تحليل طيف تقنية (LIBS) أوضحت أن عدد من الذرات مثل (الحديد، التيتانيوم، الأوزميوم، الثليوم، الباريوم، الزركونيوم، النحاس، الأكسجين، النيوبيوم، والزنك) قد ظهرت في هذه العينة. بالإضافة إلى ذلك أن أيونات بعض العناصر على سبيل المثال (Zr+3، Ti+3، Cu+3، Fe+5، Ti+6، Ba+3، S+2، Fe+2، O+2، S+5، Sr+2، Ba+2، و O+3)كانت حاضرة في نهاية هذه التجربة العملية. النتائج المتحصل عليها تعود إلى التفاعل الحاصل بين عينة البودر مع البلاستيك، ويعود الاختلاف في النتائج إلى دقة القياسات للتقنيات المختلفة.

معلومات البحث
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الكلمات المفتاحية
أكسيد البيروفكسايت، تفاعل، طريقة تفاعل الحالة الصلبة، أيونات، فترة التخزين