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Chemistry Department

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Realized by Hammoudi Aya

Preparation and characterization of a mixed oxide based on iron

Presented 13 July of 2022 Infront of the jury :

S.Hammani	M.C.A	University Blida 1	President
A.Bessi	M.C.B	University Blida 1	Examiner
N.Salhi	Professor	University Blida 1	Promoter
A.Boulahouache	M.A.A	University Blida 1	Co-Promoter

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Dedication

I would like to warmly thank my dear mother for her limitless support and love throughout the years , there is no way I can repay what have you done for me.

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Special thanks to my siblings for their beautiful hearts, for being the best brother and sister I could ever wish for ,thank you.

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Abstract

Framework guide to complete the master's thesis, the research work is dedicated to synthesizing a mixed oxide material type Bi-Fe-O through the sol-gel method. This material is calcined at 300°C for 5 hours and then characterized by numerous analysis techniques: Fourrier transform Infrared spectroscopy (FTIR), X-ray fluorescence (XRF) and X-ray diffraction (XRD), scanning electron microscopy (SEM). The median crystallite size is evaluated by the empirical law of Debye -Scherrer.

The IR spectrum indicates the presence of vibration bands characteristic of O - M (Bi -O - Fe: Bi- O; Fe - O) stretching mode.

The TGA thermogram shows the formation Bi-Fe-O mixed oxide starts over 400°C.

The XRD analysis shows the presence of only the Bi_2O_3 phase and the absence of the perovskite $BiFeO_3$ phase.

The median crystallite size estimated using the Debye Scherrer formula is around 28 nm.

(SEM) assisted with EDX Energy Dispersive X-ray spectroscopy. E illustrates the spotting of porous surface morphology with heterogeneous particles size from 5µm to 20 µm. EDX shows that a major part of the surface in the sample is covered with bismuth and iron more than oxygen. XRF analysis shows the major atomic elements with experimental percentages 68,52% for bismuth and 22,04% for iron .

Key Words: Synthesis, Photocatalyst, Hydrogen, Mixed-oxide, Sol-gel.

Résumé

Guide cadre pour la réalisation du mémoire de master, le travail de recherche est consacré à la synthèse d'un matériau d'oxyde mixte de type Bi-Fe-O par la méthode sol-gel. Ce matériau est calciné à 300°C pendant 5 heures puis caractérisé par de nombreuses techniques d'analyse : Spectroscopie infrarouge à transformée de Fourrier (IRTF), fluorescence X (XRF) et diffraction des rayons X (XRD), microscopie électronique à balayage (MEB). La taille moyenne des cristallites est évaluée par la loi empirique de Debye -Scherrer.

Le spectre IR indique la présence de bandes de vibration caractéristiques du mode d'étirement O - M (Bi -O - Fe : Bi- O ; Fe - O).

Le thermogramme TGA montre que la formation de l'oxyde mixte Bi-Fe-O débute à plus de 400°C.

L'analyse DRX montre la présence d'une seule phase Bi_2O_3 et l'absence de la phase pérovskite $BiFeO_3$.

La taille moyenne des cristallites estimée par la formule de Debye Scherrer est d'environ 28 nm.

La microscopie électronique à balayage (MEB) assistée par la spectroscopie de rayons X à dispersion d'énergie EDX. Illustrer une morphologie de surface poreuse avec des particules hétérogènes de 5µm à 20 µm. EDX montre qu'une majeure partie de la surface de l'échantillon est couverte de bismuth et de fer plus que d'oxygène. L'analyse XRF montre les principaux éléments atomiques avec des pourcentages expérimentaux pour le bismuth 68,52 % et le fer 22,04% .

Mots clés : Synthèse, Photocatalyseur, Hydrogène, Oxyde mixte, Sol-gel.

الملخص

في إطار إكمال أطروحة الما ستر، تم تخصيص العمل البحثي لتركيب مادة أكسيد مختلطة من نوع Bi-Fe-O من خلال طريقة sol-gel. يتم علاج هذه المادة عند 300 درجة مئوية لمدة 5 ساعات، تمر عبر تقنيات تحليل عديدة: التحليل الطيفي للأشعة تحت الحمراء (FTIR) وتحويل الأشعة السينية (XRF) وحيود الأشعة السينية (XRD) ومسح الإلكترون المجهري (SEM) التحليل الحراري الوزني (TGA). يتم تقييم الحجم المتوسط للكريستاليت من خلال القانون التجريبي لديباي-شيرير.
يشير طيف الأشعة تحت الحمراء إلى وجود نطاقات اهتزاز مميزة لوضع التمدد M - O) O - Fe: Bi- O, Bi -O - Fe -).
يوضح مخطط TGA الحراري أن تكوين أكسيد مختلط Bi-Fe-O يبدأ بأكثر من 400 درجة مئوية.
يُظهر تحليل XRD وجود طور Bi_2O_3 فقط وغياب طور البيروفسكايت $BiFeO_3$.
يقدر حجم المتوسط للكريستاليت باستخدام صيغة Debye Scherrer بحوالي 28 نانومتر.
المسح المجهري الإلكتروني (SEM) بمساعدة التحليل الطيفي للأشعة السينية المشتتة للطاقة EDX. يوضح SEM اكتشاف مورفولوجيا السطح المسامي بجزيئات غير متجانسة بحجم من 5 ميكرومتر إلى 20 ميكرومتر. يوضح EDX أن جزءًا كبيرًا من السطح في العينة مغطى بالبزموت والحديد أكثر من الأكسجين. يُظهر تحليل XRF العناصر الذرية الرئيسية بنسب مئوية تجريبية لكل من البزموت68,52% والحديد22,04% .

الكلمات المفتاحية: التوليف ، الحفاز الضوئي ، الهيدروجين ، أكسيد مختلط ، سول-جل.

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List of abbreviations

- **CB:** Conduction band.
- **Eg:** Energy Gap**.**
- **EDX:** Energy dispersive x-ray spectroscopy.
- **FTIR:** Fourier transform infrared spectroscopy.
- **hv:** Photon energy .
- **SEM:** Scanning Electron Microscopy.
- **t_a:** Goldschmidt tolerance factor.
- **VB:** Valence band.
- **XRF:** X-ray fluorescence.
- **XRD:** Electron X-Ray Diffraction.
- **TGA:** Thermogravimetric analysis .

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General introduction

Introduction

Energy played a big role in research and science in the past decades, it's been observed by all scientists over its various versions.

All organisms rely on energy to survive, over time scientist started their journey to create energy using physics and mathematical notions and they touched the invention of steam engines reaching the 20's century when fossil fuels were discovered such aspetroleum, coal and natural gas**[1].**

Another energy was uncovered most powerful and in the same way, the most harmful to humankind using uranium and converting it creating "nuclear energy "**[2]**.

All these sources of energy are used to generate engines and power. Electricity is the primary source of energy for it's easier use .

This resource is known as expensive to extract and convert, as well as non-renewable and non-sustainable for the ecosystem including its big contribution to global warming**[3].**

Therefore, the research begins for renewable resources of energies that are in the disposal and biodegradable will continue to rise and supersede fossil fuels before extinction, like solar, hydro, wind, geothermal and biomass**[4].** This endless supply like sun, water and wind and some sources need effort and time to domesticate**[4]**.

The research is always active to find more resources for energy that is biodegradable and renewable that has good potential to reduce sustainably carbon emissions**[5].**

Most search topic in renewable energy is biofuel to replace coal and petroleum where we encountered biodiesel made from plants also other renewable sources such as solar most abundant sources of energy along with hydro (H_2O) that will produce biofuel such a hydrogen fuel which is used in multiple industries it's a combustible element and that makes it a great fuel**[6].**

Production of H_2 can be conducted under various methods mostly through thermochemical procedures that use natural gas , organic materials, or H_2O .The H_2 extracted through the molecular hydrocarbon structures generally via vapor reforming gives out the highest percentage of H_2 **[7]** .

Also, partial oxidation is the fastest, we encounter electrolytic processes where we find the detachment of O_2 and H_2 by electrolysis of water that takes location in the electrolyze but unfortunately, the electrode corroded as some as the current is applied. We see other methods which are biological processes that utilize bacteria and microalgae .The process that we are most interested in is a photoelectric process which produce hydrogen via light energy , in this case H_2 can be obtained by using semiconductors that separate water into H_2 and O_2**[8].**

One of the materials that can be used as semiconductors are metal oxides such as Perovskites that has ability to absorb and radiate light ,one of the most prominent properties are electric , ferroelectric and catalytic properties, all due to their crystalline structure **[9].**

Perovskite oxides are presented against high Ultraviolet ray radiation activating their semiconductor properties which is the most important parameter in producing hydrogen through photodegradation of H_2O, playing the role of photocatalyst **[10].**

Production of hydrogen fuel that produces electricity as a byproduct is an eloquent renewable resource to supersede fossil fuels, through photodegradation of water via photocatalytic reaction.

In this framework , the synthesize of mixed oxide based on iron and bismuth via sol-gel method.

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Theoretical study

1-Catalyst

The past decades have been revolutionary to the synthesis and application of catalysis around 85 -90 percent use catalytic processes in chemical reactions, that dueto alternative, energetically promising mechanisms to the non-catalytic reactions, consequently facilitating processes its carried beneath industrially practicable requirements of pressure and temperature**[1].**

Catalyst its chemical material that takes the role to accelerate the reaction by forming bonds with the reactant without causing disruption or change to any reactant agent where they react to synthesize the product with less energy. They enhance the rate of the reaction that is established by an energetic barrier, which needs to be overcome to transform reactants to products. This energy is called activation energy , the catalyst will maintain the same physicochemical characteristics , one of the identifying properties is its crystalline and amorphous structure**[1].**

Catalysis is outstanding significance as it impacts our regular life, four primary sectors of the global economy; petroleum and energy production, chemicals and polymer production, nutrition industry, and pollution control involve catalytic procedures shown in fig1.

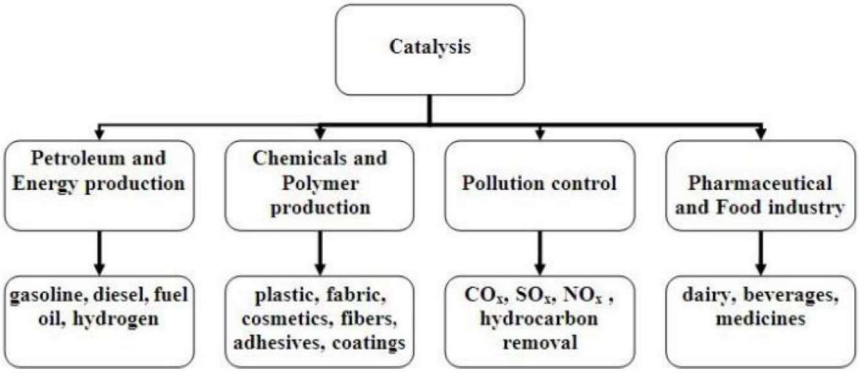

Figure 1: Primary sectors of the global economy that affect catalytic processes [2]

Catalysis concerns comprehending the thermodynamics, kinetics, electronic interaction, crystal structure, reactor layout and process evolution of a catalytic process. The catalyst will just change kinetics and thermodynamics side of the reaction will not in contour any changes. Thermodynamics of a reaction It is an interdisciplinary area implicating contribution from chemists and material scientists for the victorious execution of the whole process initiating from the trial of catalysts to final utilization in a chemical reactor**[1].**

1.1. Catalytic reactions

A catalytic chemical reaction is only possible when an additional slight amount of chemical substance that raises the rate of accomplishment of a chemical equalizer despite the substance itself does not undergo any chemical change, the reaction its titled a catalytic reaction**[3].**

In an automatic reaction, the catalytic reaction starts by bonding the reactants A and B to the catalyst. Therefore, the formation of this complex is exothermic, and the energy is reduced. There is a reaction among A and B while they are attached to the catalyst exterior only contact. This phase is associated with activation energy,nonetheless, it is remarkably more subordinate to uncatalyzed reaction. Ultimately, the product breaks from the catalyst in an endothermic phase, catalytic reactions frequently operate at elevated pressure for the case of heterogenic phases**[3].**

We conclude that the catalyst improves reaction rate and delivers alternative mechanisms involving another evolution state of with less activating energy. Thereby, the activation energy of the catalytic reaction is reduced compared to the uncatalyzed reaction fig 2.

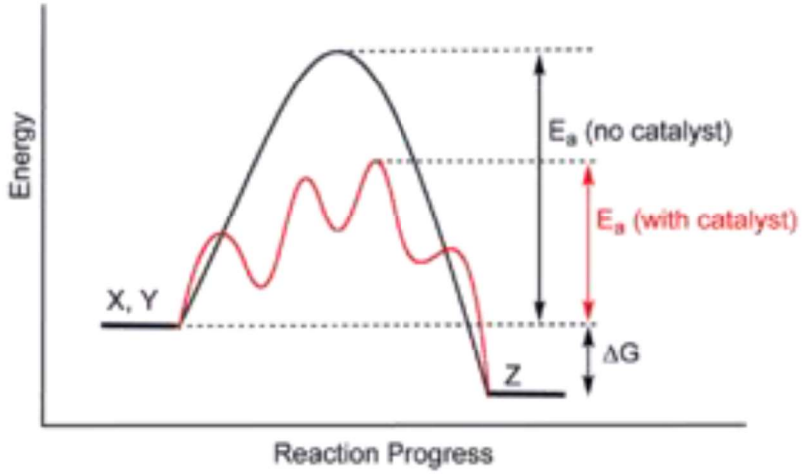

Figure2: Effect of catalyst presence on activation energy and reaction progress [4]

1.2. Classes of catalytic reactions

Catalysts arrive in numerous shapes and are usually utilized in polycrystalline form. The reactions that ensue in specific areas in the characteristics of each microcrystal, varyingfrom atoms and molecules to enormous structures such as zeolites or enzymes. In addition, they may be utilized in various surroundings like solutions , gases or at the exterior of solids**.**Then, to understand this process we can examine the reaction on the surfaces of the crystalline materials**[5].**

1- Biological catalysis

Known as enzymes this innately emerges in alive organisms. The reactant in this reaction its entitled a substrate and an enzyme reverse it to another molecule resultingin the preferred product. They are primarily proteins **[5].**

2- Heterogeneous catalysis

Both the catalyst and reactants are independent phases in this type of reaction.Usually, everything will be either as gas or in a liquid phase. We encounterseveral hydrocarbon conversion reactions like cracking, reforming, dehydrogenation and isomerization. We find the contact process using V_2O_5 or Pt as a catalyst to produce sulfuric acid, the Haber process using Fe, K_2O and Al_2O_3 as a catalyst to produce ammonia and catalytic hydrogenation using Ni, Pd, or Pt as a catalyst to be applied partially hydrogenated oils for margarine**.** Heterogeneous catalysts, particularly solid catalysts, dominate the industrial catalytic processes **[5].**

3- Homogeneous catalysis

The catalyst is in the same state as the reactants in this catalysis type . Usually, both reactant and catalyst will be either as a gas or in a liquid phase. Number of commercially achievableprocesses have been created in current years which operate as hydroformylation catalysts to produce aldehydes using Rh/PR_3 precursor , adiponitrile process to produce nylon using Ni/PR_3 complexes **[5].**

1.3 Requirements of catalyst

Catalysts can be metals, oxides, salts, or other types of material. Solid catalysts come in a different form and can be loose or small particles on support. The support could be a porous powder, such as the ceramics utilized in vehicle systems, clays or zeolites could also be catalysts **[3] .**

We listed the following requirements [3]:

- Elevated activity.
- Elevated selectivity.
- Sufficient thermal stability.
- High mechanical strength.
- Elevated corrosion resistance.

2-Photocatalysis

In search of a new alternative form of renewable energy, decrease CO_2 emission, one of the main streams in the universe for renewable energy sources is sun rays that are supplementarily acceptable to provide energy and replace nonrenewable once. This comes through conversion of ultraviolet light to hydrogen as long-term **[6].**

Photocatalysis is the subordinate branch of catalysis that explores the chemical reactions that are initiated by the presence of light. The light activates the catalyst and consequently evokes chemical reactions. Some standard photoreactions are oxygen reduction and oxidation of organic compounds, while the multiple known photoreactions are photosynthesis**[7].**

In photocatalysis, a semiconductor material could be employed to execute a chemical reaction induced by photons as an energy source. An ideal photocatalyst is photostable, chemically inactive and available at a reasonable cost. Although a category of semiconductors is now obtainable**[7].**

Photocatalysis could be distributed into two main methods :

1. Thermochemical methods

Radiation containing sunlight of wavelengths from 280 to 4000 nm is transformed into thermal energy that provokes a chemical reaction, this process is encountered in steam reforming of methane, fulfilling the next conditions **[7]:**

- The thermochemical reaction must be endothermic.
- The method must be cyclic and with no side reactions .
- The maximum usage of solar energy.
- The very slow back reaction .
- The products of the photo-chemical reaction are easy to stock and transport .

2. Photochemical methods

Photons are instantly absorbed by reactants or a catalyst generating a reaction, in this procedure, a semiconductor is activated with ultravioletlight, and a photoexcited electron is elevated from the VB to the CB, assembling an electron/hole pair (e^- and h") , this pair has the capability to provoke reduction and oxidation reactions on the photocatalyst surface**[7]**.

semiconductor + $h\nu \rightarrow e^- + h"$

2.1.Photocatalyst

A substance that could absorb light, such as semiconductor , will induce chemical conversions of the reactants , constantly reaching with them into intermediate chemical exchanges and regenerating its chemical composition after each cycle of such interactions. The physicochemical properties of the material are required foracceptable performance, and these are usually inducted by the photocatalyst nature(composition, size, shape, morphology and the source of the material) **[8].**

The difference in conceptions of catalytic and photocatalytic reactions: A catalyst contains active sites of which a substrate is converted into a product, while no active sites are present on a photocatalyst, but the photo activity is created via UV lights at the exterior of the semiconductor **[9].**

2.2.Photocatalytic mechanism

In this type of reaction, semiconductors absorb ultraviolet light, it will undergo a redox reaction, in this reaction we will find electron excitation from the VB to theCB , leavinga hole(h) in the valence band. **[10].**

Photogenerated electrons and openings are remoted through an electrical field and move to the outer layer of semiconductor particles. The photogenerated openings have powerful oxidizing properties and might also oxidize supplies adsorbed on a superficial degree or arrangement of the semiconductor **[10].**

The conversion of energy can only happen if photons have the equivalent energy as bandgap (the difference between CB and VB), as for the electron will take apart in the reduction to produce hydrogen, or other reduction reactions that involves electrons, also we find that the holes will take part in degradation of water, also other organic compoundsdepend on the purpose of the photodegradation**[10].**

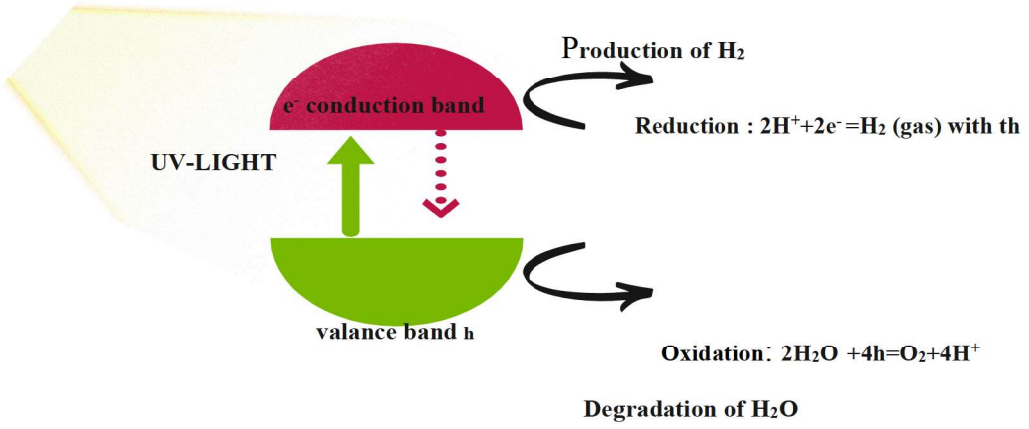

Figure3: Oxidation and reduction in semiconductors

2.3.Photocatalysis application

Photocatalysis is nowadays one of the considerable vibrant interdisciplinary study areas and have been analyzed from the perspective of catalysis, photo/electrochemistry, inorganic, organic, polymer, and green chemistry, a numeral of various procedures ranging from H_2O treatment and air cleaning to disinfection and anti-tumoral applications, and the production of hydrogen fuels from water splitting and atmospheric gases to selective organic synthesis and metal recovery. That may interest different industrial applications **[11]**

2.4. Parameters in photocatalysis

Many factors take place affecting photocatalysis reaction, these factors will influence catalyst properties such as the charge, the absorbing material, defect, composition also conditions such as pH and solvent, the concentration of reactants, adsorption, and product examination of reactants oxygen concentration and utilized light source (wavelength, intensity, distance)**[12].**

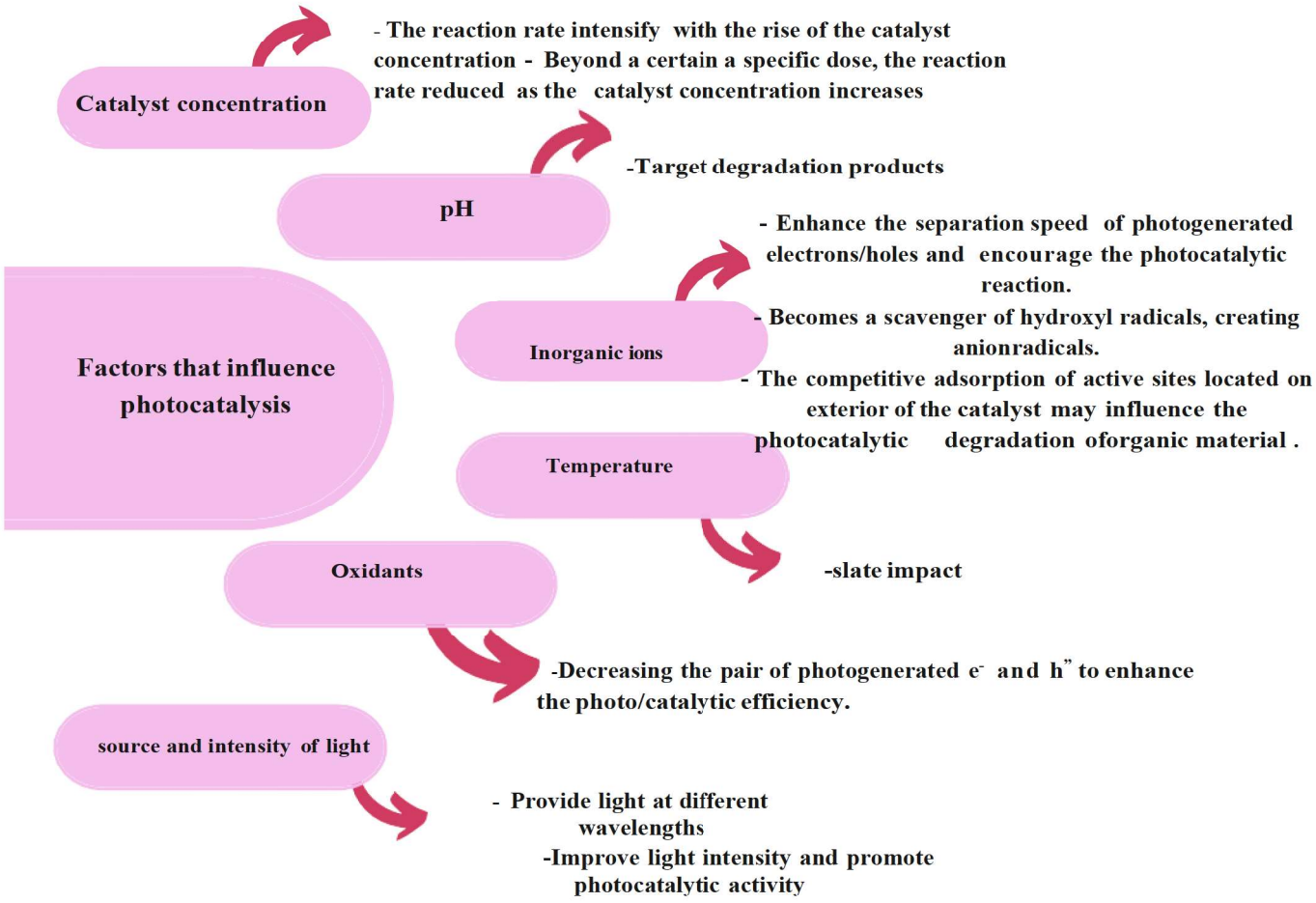

Figure 4: Influencing factors on photocatalysis[12]

3-The metallic Oxides

Oxides have permitted the evolution of catalysts with high selectivity and with new preparation procedures. The execution of a solid catalyst be dependent on its grain size, preparation **[13].**

Oxide catalysts are applied in many of industrial catalytic processes, these oxides show distinct properties such as acidity, basicity and redox features, when transition metal ions are present that will give out catalytic properties. They are also the foundation for metallic catalysts, such as perovskites with the structure **ABO_3** and **spinals** with the form of **AB_2O_4**, pyrochlores with the structure **$A_2B_4O_7$ [13].**

The core catalytic domains contain acid/base catalyze, photocatalysis, pollution, and biomass conversion **[13].**

3.1.Types of Oxides

Oxides can diverge into the subsequent classes founded on the metal valency **[14]:**

- Simple Oxides: it can be one metal or semi-metal and oxygen. this Oxides can carry only several O atoms that due to elements vacancy .

- Mixed Oxides: Formed after simple Oxides are combined. These 2 simple oxides could be of the made of the equivalent element or not .

Oxides can be split into the subsequent types due to their metallic properties **[14]:**

1- Metallic
2- Basic
3- Amphoteric
4- Non-metallic
5- Acidic
6- Neutral

3.2.Oxide preparations

The broad topic of metal oxide syntheses of bulk and supported oxide catalysts. It corresponds to 3 main areas **[15]:**

1) Simple metal oxides
2) Mixed oxides
3) Supported metal oxides.

A specific industrial procedure for manufacturing catalysts includes **[15]:**

- Precipitation at a specified pH or another synthesis process (sol-gel, solid-solid, flame hydrolysis, vapor deposition)
- Hydrothermal transformation
- Decantation, filtration, centrifugation
- Calcination, activation, reduction.

For simple oxides, it's vital to monitor catalyst with solid form , its structure , textural properties,besides thermal procedures, doesn't permit satisfactory regulation of these parameters, 3 main preparation courses grounded on different reactions between H_2O or an (organic/inorganic) solvent and precursors are usually employed, precipitation, sol-gel .This type of oxides can be utilized as a support for an active phase for a lot of chemical reactions, they can also be utilized as a catalyzer but because of superficial properties, acid base, they are not always optimized for the reaction **[15].**

The mixed oxide catalysts are prepared through co-precipitation of 2 or more salts in solution at a particular pH, surveyed by the classical heat treatments. We can also proceed by solid/solid contact amongst both salts at high temperatures **[15].**

4-Perovskite

Perovskite are appealing compounds for various applications owing to their enormous number of compounds .They display physical individualities have been used as catalysts in numerous reactions due to their elevated sensitivity, exceptional extended term stability as well as anti-interference capability**[16].**

Depending on Perovskite-phase oxides particular variety of properties became functional for diverse applications they are recently used in electrochemical distinguishing of alcohols as well for glucose, H_2O_2 , gases and neurotransmitters, photodegradation of water, and another type is perovskite showed efficient indispensable properties for photo-voltaic solar cells**[16]**

4.1. Structure

The main form of perovskite is ABX_3 for all perovskite compounds , A and B are ions , X is often oxygen or large ions such as halides, sulfides and nitrides .Perovskites exist in 3 types; the firstly contains electrons, the second holds delocalized energy-band states, and the final type can be a combination of the first and second types**[17].**

Perovskite with formula ABO_3 is a very significant type that is an efficient material that exhibit a selection of stoichiometries and crystal structures. Becauseof the structural features, they can house around 90 percent of the metallic materials of the periodic table that is positioned at the A / B positions without destroying the structure, offering a way of correlating solid-state chemistry to catalytic properties**,** The A and B can be replaced by any metal/semimetal from the periodic table**[16].**

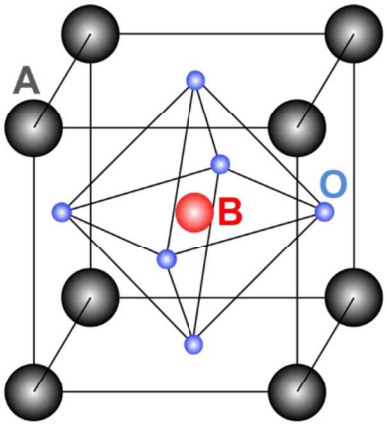

Figure5: Perovskite type ABO_3 [18]

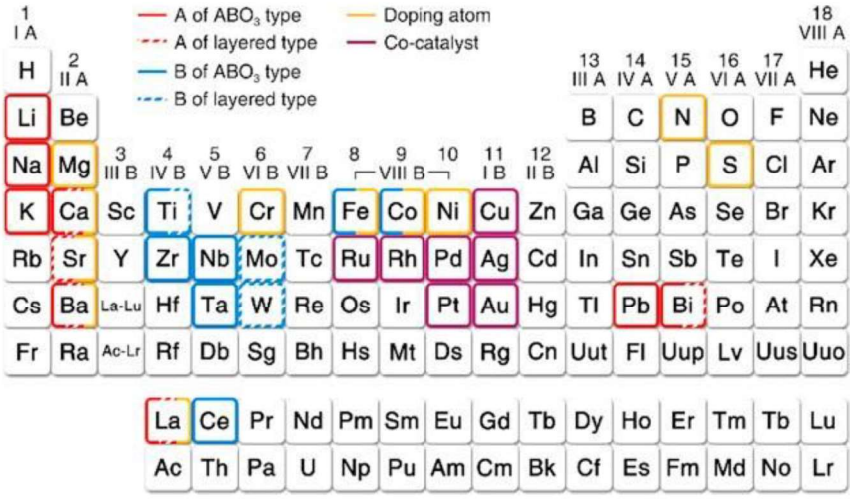

Figure6: Perovskite A and B elements in the periodic table [19]

The ABO_3 crystallographic structure with the cubic form gets the most attention, this variation is seen in this form due to the real measure of a distortion degree of ideal perovskite , so the value of t_a tends to unity as the structure approaches the cubic form, 2 necessities should be fulfilled for perovskite establishment **[20]:**

1. Electroneutrality : The structure have neutral balanced charge, A and B ions charge combained is equivalent to the full charge of the oxygen ions.

2.Ionic radii requirements: r_A superior then 0.090 nm and r_B superior then 0.051 nm, and the t_a must be within the range values $0.8 < t_a < 1.0$.

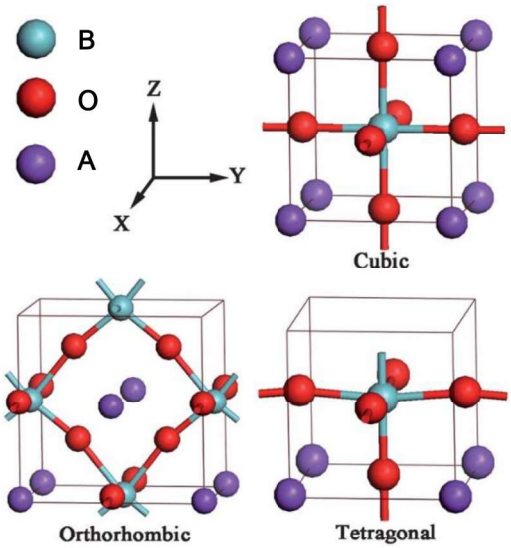

Figure 7: Different structures of perovskite [21]

4.2. Properties

Inspired by the ample properties and their characteristics of perovskite oxides, analysis of this materials largely focused on altering properties to enhance catalyticproperties, such as compositional structure and defects engineering. Herein, the figure below is recapitulating the major characteristics of perovskite oxides that aid the catalytic process **[22]:**

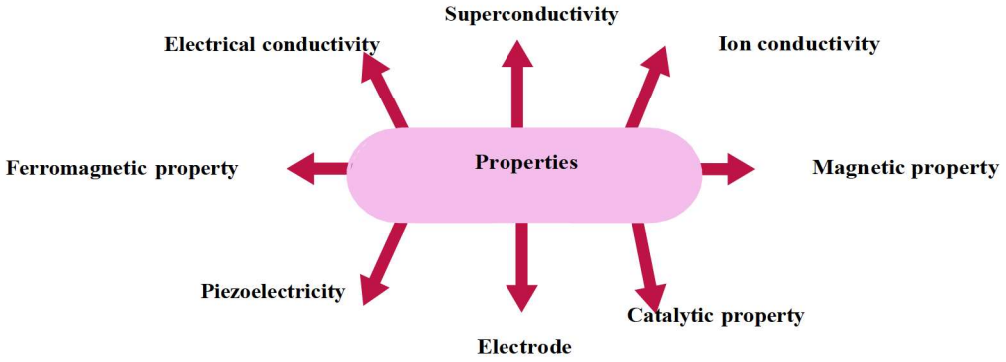

Figure 8: Properties of perovskite

Most important properties of perovskite oxides :

1.Dielectric properties

There are some properties innate dielectric properties like ferroelectricity, piezoelectricity, electrostriction and pyroelectricity. One of the vital characteristics of perovskites is ferroelectric actions **[23] .**

2. Electrical conductivity and superconductivity: the clear properties of perovskites is superconductivity, as well as exhibiting high electronic conductivity**[16].**

3.Catalytic activity and chemical stability : Perovskites present outstanding catalytic activity and elevated chemical stability. Therefore Perovskites can act as automobile exhaust gas catalysts, intelligent automobile catalysts, cleaning catalysts, etc., for various catalytic environmental reactions. It was stated in the literature this materials containing elements like Mn, or Fe display tremendous catalytic activity concerning the decomposition of NO_X at elevated temperatures, this last on is the hardest reaction in the catalysis ($2NO \rightarrow N_2 + O_2$) **[22].**

5. Catalysts preparation

As mentioned, catalysts have become a vital aspect of today's chemical reaction, they take part in most products to be utilized in our daily life. How the different catalysts perform is directly correlated with the heterogenic catalyst properties such as porosity, size, and surface area. These properties are linked, to some extent, with the methodology used for the preparation of the materials. Thus, different approaches will provide different final properties and therefore, different catalysts to be used in different applications. A description of some of the various techniques will be presented in this subsection to show these methodologies and the main effect they can have on the different materials **[24].**

Preparation of catalysts could be divided into 2 categories which are: first solid-state procedures and the perovskite oxides by this technique are synthesized at high temperature or by mechanical activation at lower temperatures **[25]** and for the second category that is wet chemical procedures, where we find multiple methods to prepare the desired catalysts ,some of them are the hydrothermal, sol-gel method, co-precipitation, impregnation, ion exchange**[26].**

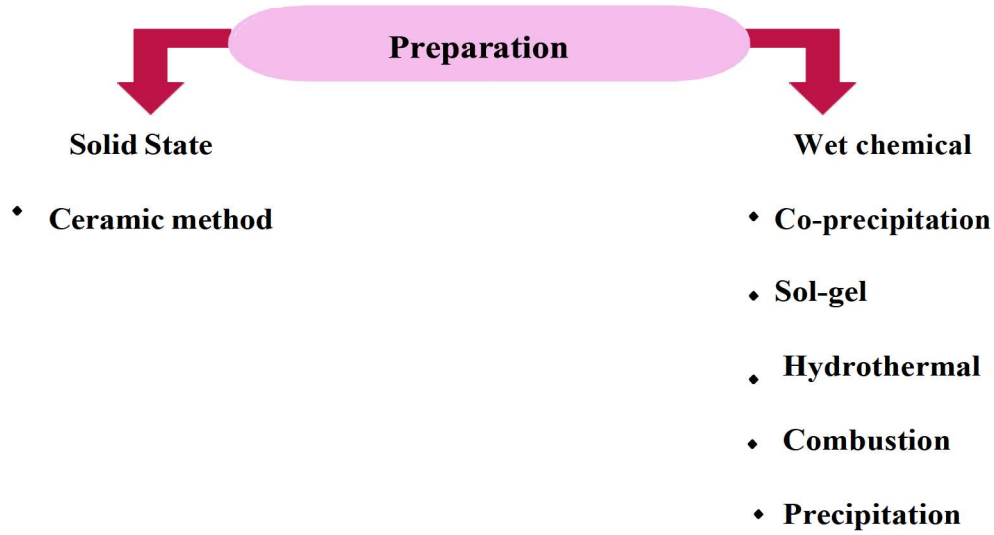

Figure 9: Preparation methods of catalysts

6. Procedures

6.1 Solid-state method

Ceramic method

This method happens at elevated temperature with 2 non-volatile solids that will produce the desired product, it also can be utilized in the preparation a complete variety of materials comprising mixed metal oxides **[27].**

- **Disadvantages [27]**

1. High temperatures are generally required (500-2000°C) because it takes a significant amount of energy to overcome the lattice energy so a cation or anion can diffuse into a different site.
2. The anticipated compound may degrade at elevated temperatures.
3. The reaction may proceed very slowly, but boosting the temperature fasten the reaction since it increases the diffusion rate.
4. Generally, solids are not elevated to their melting point, so reactions take residence in the solid-state.

6.2 Wet-chemical methods

Co-precipitation

The co-precipitation technique is generally used to produce two component-based catalysts (for example, metal-based catalysts). This technique has very closely related properties to the precipitation technique, and it is generally introduced as one subsection. This methodology differs from precipitation mainly since it is not possible to achieve homogeneity when precipitating one solute. The final composition and structure of the catalysts or catalyst precursors will depend, as before, on several factors, such as pH, temperature, mixing rate and the solubility among the two metal-based reagents. This solubility, between the reagents and the liquid, will lead to different types of catalysts, with different properties and purpose . Therefore, the same metal reagents could lead to two very different catalysts with different applications based on the solution liquid **[28].**

Sol-Gel method

This method is very commonly used to prepare a carbon-based component, metal oxides, silicon-based materials, and zeolites. This method consists mainly of the transformation of monomers or solids particles that are in a liquid (sol) into a gel, which can contain both liquid and solid phases. To obtain the final catalytic materials, removal of the liquid phase is required. This can lead to two main types of products, an aerogel or a xerogel, where the main difference can be seen in the shrinkage experience by the materials and this shrinkage is related to the type of employed drying process **[29].**

The advantages of this methodology reside in the process simplicity that ccould be utilized to produce the desired catalysts, and the structure can be tailor-made based on the applications. Some applications are within the area of energy, aerospace engineering, textiles, and solar cell application **[29].**

- **Advantages [30]**

1. It produces a thin coating to ensure excellent adhesion between the substrate andthe top layer.
2. It has the power of starting at low temperatures, between 200°C and 600°C.
3. A easy, inexpensive and efficient method to produce high-quality coverage.
4. Synthesizes products with elevated purity due to the organometallic precursor of ceramic oxides can be dissolved in a specified solvent and hydrolyzed in soil and subsequently a gel, the composition can be highly controllable.
5. Use organic solutions that can be non-toxic.

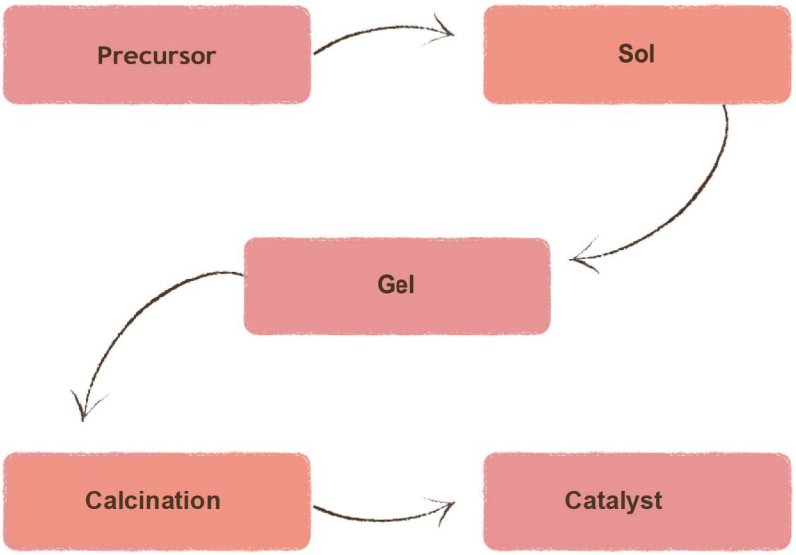

Figure 10transforms: sol-gel process

7.Perovskite $BiFeO_3$

The paradigm of single-phase ferroic material has potential applicationsin information storage, sensors, and actuators. This perovskite has a rhombohedral R_3c crystallography display magnetoelectric combination at temperature between 20-25°C **[31].**

However, its low remanent magnetization and relatively important leakage current are the main limitations for possible applications. **[32].**

BiFeO3 shows Ferroelectric/Ferromagnetic properties simultaneously and hence is a multiferroic material**[33]** also has high conductivity, which may originate from uncertain oxygen stoichiometry, and high defect density**[34].**

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Experimental part

1.Precursor preparation

The method utilized to prepare the mixed oxide iron-based is wetchemical method using sol-gel using propionic acid as a solvent due to its high performance.

The table below lists the properties of each salt utilized for this synthesis.

Table1: Physiochemical properties of precursor salts

Name	Chemical formula	Molar mass	Fusion °C	Physical state	Toxicity	Purity
Bismuth nitrate	$Bi(NO_3)_3.5(H_2O)$	485,07g/mol	33°C	White crystalline powder	Irritant	98%
Iron nitrate	$Fe(NO_3)_3.(H_2O)_9$	404 g/mol	47°C	Pale violet	Oxidizer, Irritant	99%

Table 1: Physiochemical properties of precursor salts

1.1 Masse calculation

To obtain 15 g of BiFeO3, we calculate precursors salt masses that needed to obtain such results :

$$Bi(NO_3)_3.5(H_2O) + Fe(NO_3)_3.9(H_2O) \xrightarrow[\text{Calcination at 300°C}]{} BiFeO_3$$

- **Masse of Bismuth nitrate**

$M_{Bi(NO3)3.5(H2O)}$= 485,07 g/mol

M_{BiFeO3}= 312,82 g/mol

485,07 g ⟶ 312,82 g

X ⟶ 15 g

$X=\frac{485,07*15}{312,82}=23,26$ g

- **Mass after purity percentage consideration**

$X'=\frac{23,26*100}{98}=23,78$ g

The final mass of Bismuth nitrate to obtain 15g of the product is **23,78g.**

- **Mass of Ferric nitrate**

$M_{Fe(NO3)3.9(H2O)}$=404 g/mol

M_{BiFeO3}= 312,82 g/mol

404 g ⟶ 312,82 g

Y ⟶ 15 g

$Y=\frac{404*15}{312,82}$=19,37 g

- **Mass after purity percentage consideration of ferric nitrate**

$Y'=\frac{19,37*100}{99}$= 19,57 g

The final mass of ferric nitrate to obtain 15g of the product is **19,57g**

1.2 Synthesis of Mixed Oxide Bi-Fe-O

After the calculation, we will now proceed with the synthesis process of mixed oxide Bi-Fe-O via the sol-gel method.

Firstly, we'll start by drying the starting materials , for the case of Bismuth Nitrate that is placed in a desiccator overnight to remove the maximum of humidity from our hygroscopic precursor that was absorbed from the environment, meanwhile, we placed ferric nitrate inside the thermostatic oven at 37°C overnight.

After the drying, we begin by placing each of these precursors in individual beakers with propanoic acid $CH_3CH_2CO_2H$ as a solvent on a heated magnetic stirrer for 10-20 min and with the temperature of 100°C , until we obtain the total dissolution of the salt.

Once we obtained uniformed solution with no particles left, we add one of the solutions to the other .

We keep the same condition until we noticed small emission of red vapor of NO_x .

We start stirring for 2h while the heat between 200°C and 250°C combined with the vigorous stirring.

After a while we witness the mixture turning into a slightly looking gel, we set aside 1g slightly from the mixture into a porcelain crucible to be tested for the TGA test. Then we continue stirring under heat until we obtain at the end brown-colored gel.

We remove our gel from the beaker into another porcelain crucible where we take it to the next step which is thermal activation using calcination that will action in a muffle furnace type NABERTHERM at 300°C.

We started our oven at 50°C and the temperature increased by 5° per min to reach 300 °C and left at the same temperature for 5h.

Then we retrieve our product from the furnace , then is crushed using a mortar to obtain fine powder.

Ferric nitrate

Dissolution of 19,57 g in Propionic acid

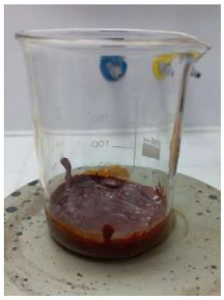
Salt dissolution

Bismuth nitrate

Dissolution of 23,78 g in Propionic acid

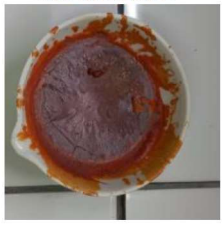
Salt dissolution

Sol mixture



mixing both solutions in a beaker under heat and stirring

Gel mixture



The gel mixture is poured into a porcelain crucible

Fine powder after being crushed

Calcination at 300°C

Figure 3: Preparation protocol of mixed oxide

Conclusion

we prepared the mixed oxide Bi-Fe-O, based on bismuth and iron utilizing a sol-gel method calcinated at 300°C for 5h . It will be analyzed by different characterization methods that are SEM, FTIR, XRF, XRD,TGA,EDX.

Result and discussion

1- Characterization of Mixed oxides Bi-Fe-O

1)Fourier transform infrared spectroscopy (FTIR)

The FTIR spectroscopy analysis was performed in order to examine the chemical bonding between different metals and oxygen. The material was analyzed in the IR domain in the 400-4000cm^{-1} range using a Perkin Elmer GXFT-IR 2000 spectrometer. IR spectrum of the synthesized material is represented in the figure below.

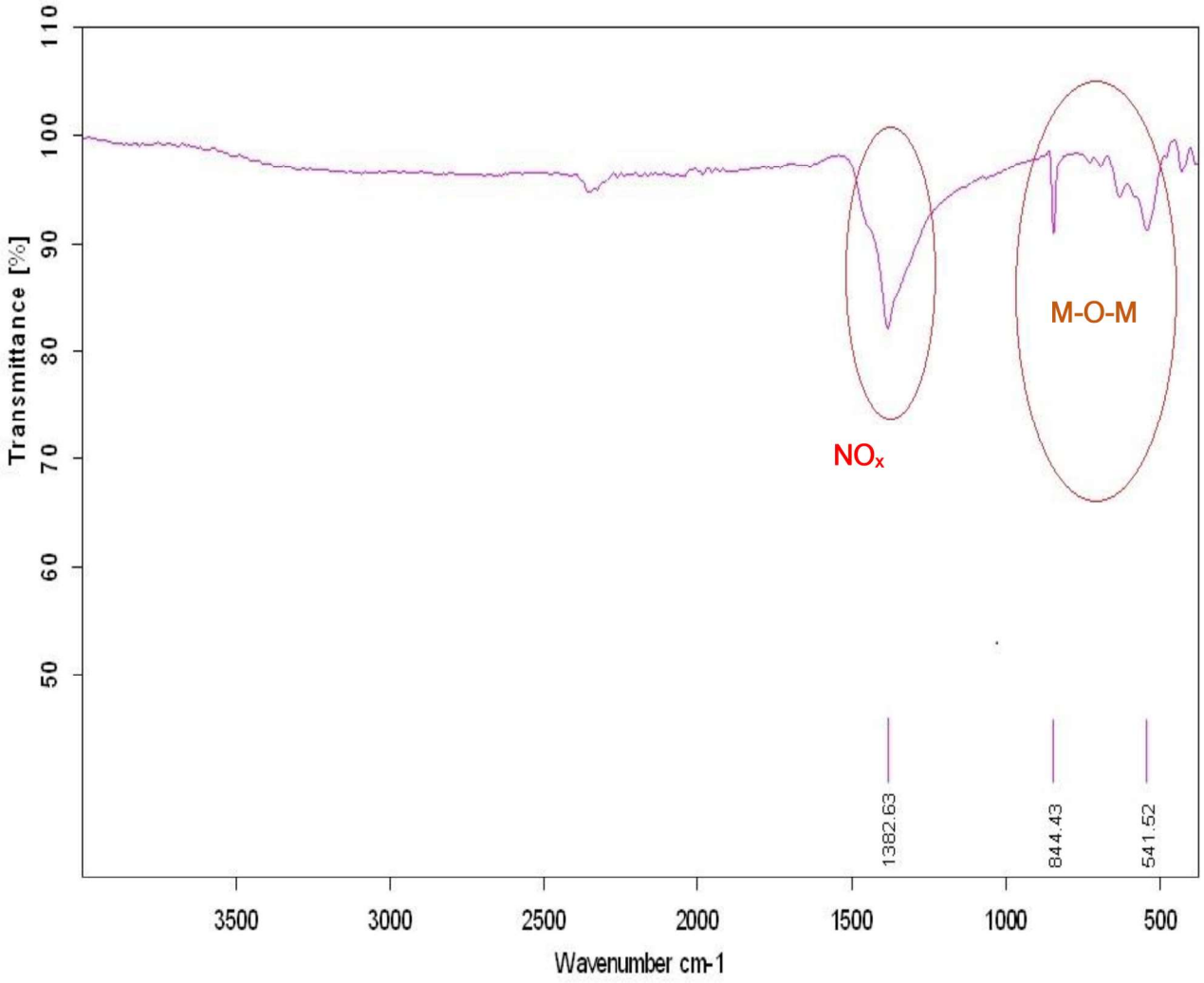


Figure 1:FTIR spectrum

The IR spectrum represents two bands in the 400-1000cm^{-1} range located at 541,52 cm^{-1} and 844,43 cm^{-1} respectively and are related to the metal-oxygen stretching vibration mode M-O-M (Fe-O, Bi-O, or Bi-Fe-O) **[1].** A strong and slightly wide band at approximately 1382,63 cm^{-1} is due to the existence of trapped nitrates as reported in previous works**[2]**.

2) Thermogravimetric analysis (TGA)

The thermogravimetric analysis is an analytical procedure used to distinguish a material's thermal stability through observing the weight transformation that ensues in the material while temperature increases at a steady rate [3].

Thermogravimetric TG experiment of gel was performed on SDT-Q600, thermobalance apparatus. 50mg of a non-calcined sample are heated from 25 to 600°C under an inert nitrogen atmosphere with a flow of 100 $ml.min^{-1}$ and a growth of temperature of 10°C/min $10°C.min^{-1}$.

Thermogram of mixed oxide Bi-Fe-O gel non calcinated is represented in the figure below :

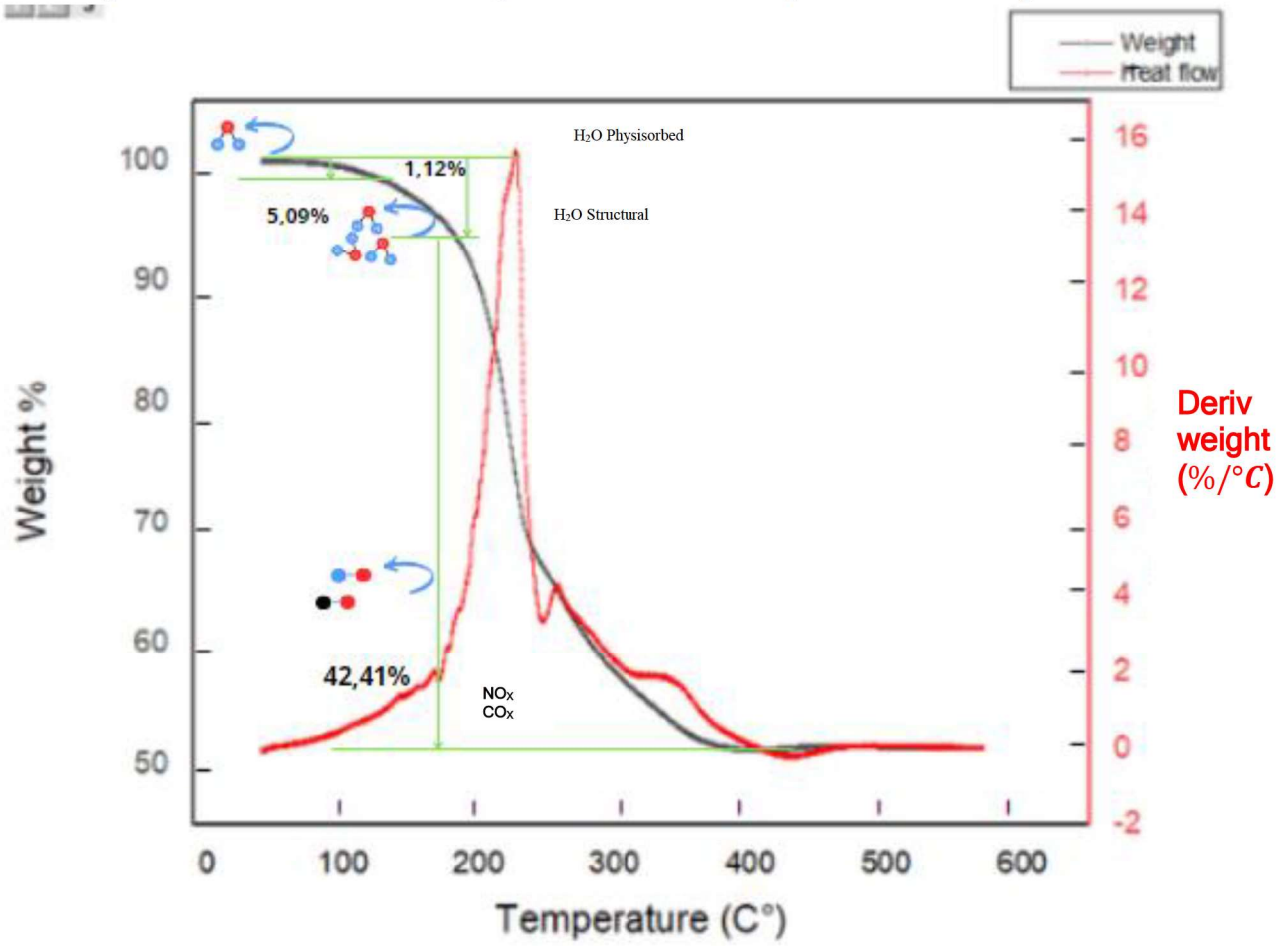


Figure 2: Thermogram of Bi-Fe-O gel

As can be seen, the thermogram of the mixed nitrates salts for Bi-Fe-O gel proceeds via three stages of Weight loss. The first progressive mass loss of 1,12% in the temperature range of 40 to 100°C is related to the release of physisorbed H2O. The dehydration of crystallization water of both bismuth and iron nitrate occurs at 100-183°C with incremental weight loss of 5,09%. A third weight loss of 42,41% progressive then brutal in the 183-400°C range , could be assigned to the simultaneous decomposition of both bismuth and iron nitrate in the form of NOx, Cox release. Over 400°C, thermal stability is observed indicating the start of the bismuth and iron oxides formation.

3)X-ray diffraction (XRD)

The XRD analysis gives out specific information, (crystalline composition, structure and particle size) **[4].** Structural characterization and the mediate crystallite size are determined by D_2 PHASER-BRUKER X-ray diffractometer using CuKα radiation (1.5406 Å) source. The data are collected at a 2θ range of 10-80° range. The XRD analysis of the Bi-Fe-O sample calcined at 300°C is represented in the spectrum below. The different phases are confirmed by the JCPDS cards.

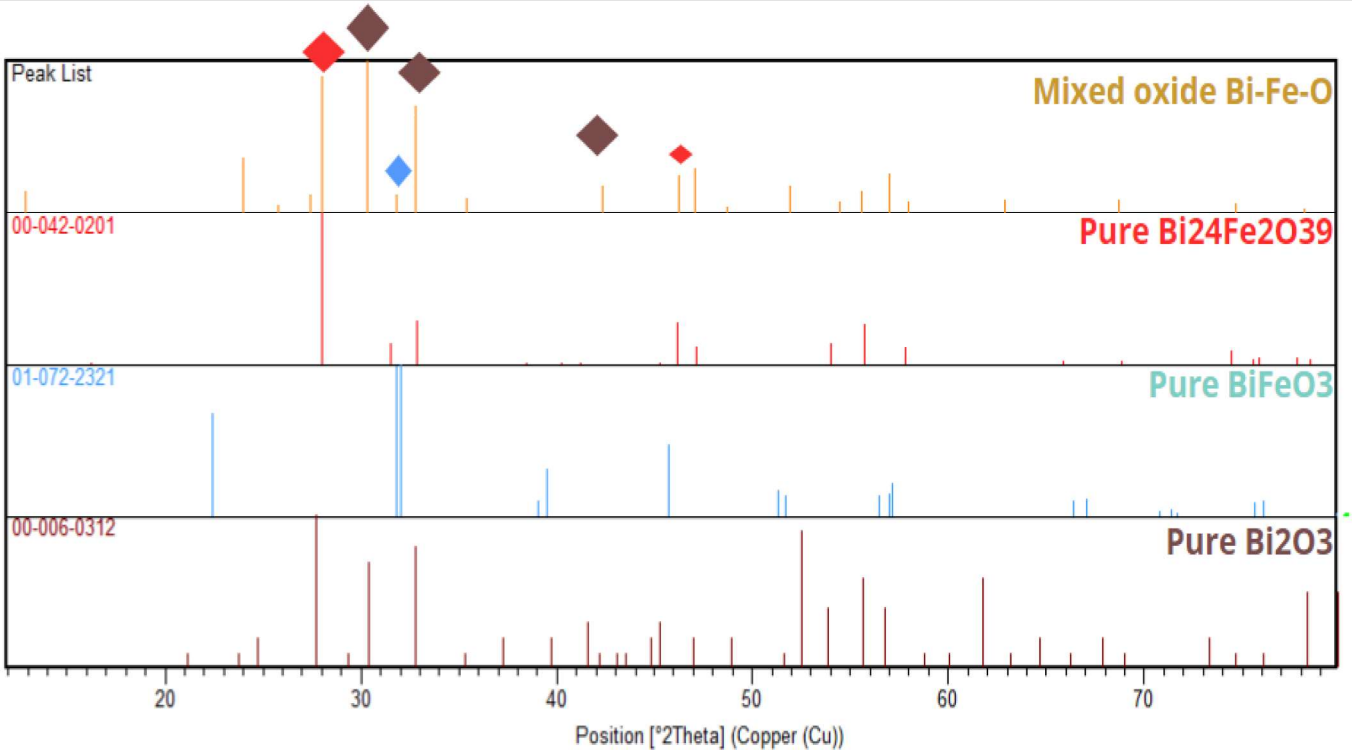


Figure 3: Spectrum XRD of the structure Bi-Fe-O

The table below represents 2θ of each Bi-Fe-O pure sample

Table 1: 2θ values of Bi-Fe-O pure material

Phases	Value of 2theta (°)	JCODS cards
$Bi_{24}Fe_2O_{39}$	27,97 -31,52 - 32,83 -46,18- 55,73	00-042-0201
Bi_2O_3	27,68 - 30,37 - 42,19- 32,77- 47,04 - 52,55 - 55,52 - 61,80	00-006-0312
$BiFeO_3$	22,38 - 31,72 - 32 - 45,69	01-071-2494

We notice the presence of peaks at respectively **2θ**= 30,29°; 32,78° and 47,05° indicating the presence of Bi2O3 phase. The $Bi_{24}Fe_2O_{39}$ phase is also identified showing peaks at **2θ**= 27,97° and 42,19°.

At our operating conditions, the DRX analysis result shows the absence of the pure perovskite $BiFeO_3$ phase and the presence of the mixture of Bi_2O_3 and $Bi_{24}Fe_2O_{39}$ oxides.

3.1.Calculation of crystallinity size of mixed oxide Bi-Fe-O

The Crystallites size of the Bi_2O_3 phase was calculated using the Debye-Scherrer formula **[5]** from the full width at half maximum (β, rd.) of the intense XRD peak and then compared to that one obtained via software X'Pert HighScore Plus software.

Figure 4 represents the aggrandizement of the most intense peak of Bi_2O_3 in the XRD spectrum.

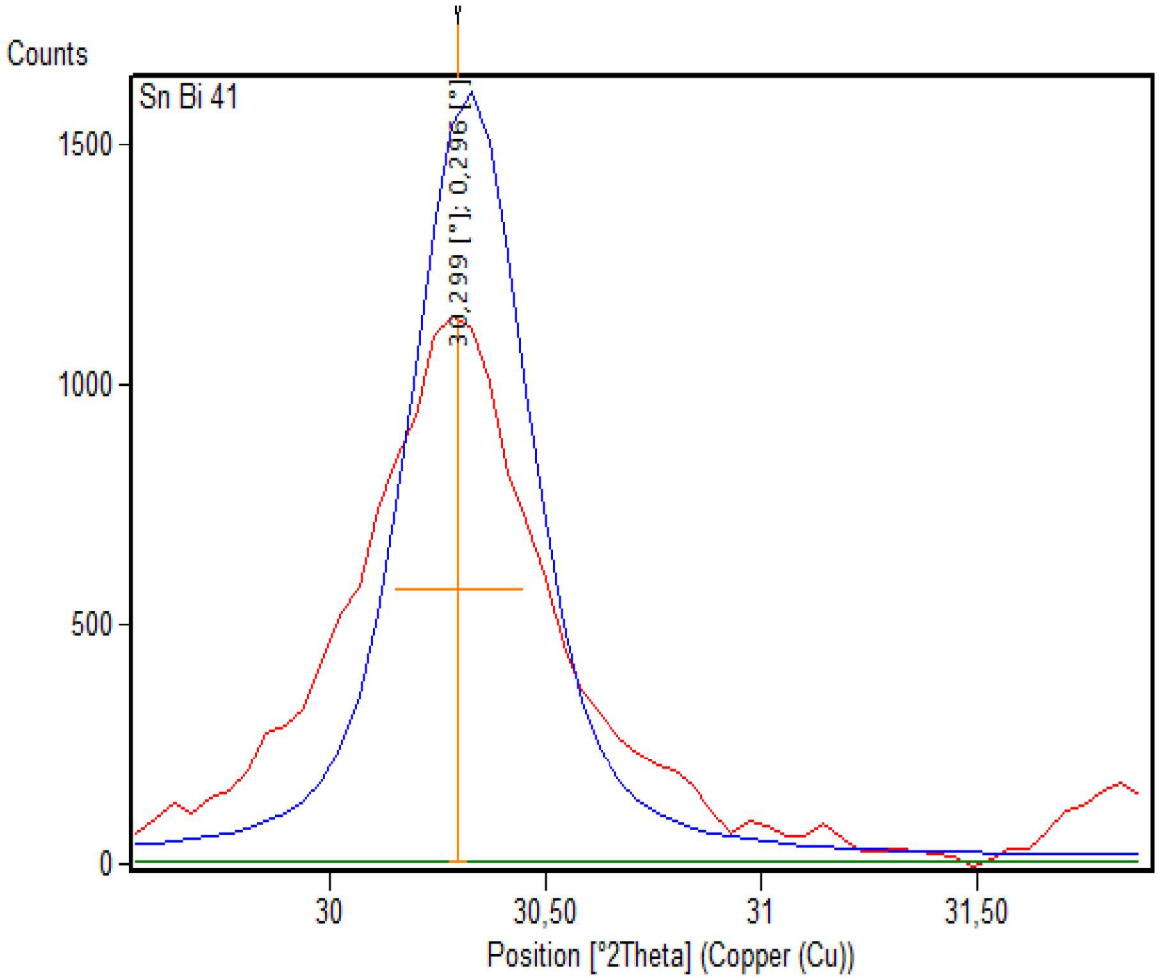


Figure 4: Aggrandizement of the most intense peak at 2θ=30,29°

We will calculate the median crystallite size through the Debye-Scherrer Law below **[5]** :

$$D = \frac{K * \lambda}{\beta * \cos\theta}$$

K: The particle shape factor, *k*= 0.9.
λ : Monochromatic wavelength of copper , λ= 1,54178 A° = 0,154175 rad.
β: The half-width of the most intense peak.
θ: Diffraction angle of the most intense peak.

2θ = 30,299°.
$\boldsymbol{\theta} = \frac{30,299}{2} = 15,149° \longrightarrow \boldsymbol{\theta}$= 0,2644 rad .
β = 0,296° = 0,00516 rad .

$$D = \frac{0,9 * 0,1541}{0,00516 * \cos 0,2644}$$

$$D = 27,82 \text{ nm}$$

Table 2 illustrates the median crystallite size obtained through the Debye-Scherrer equation and X'pert HighScore Plus software.

Mixed Oxide	Debye-Scherrer equation	Software	ΔD
Bi-Fe-O	27,82 nm	28,28 nm	0,46nm

The median crystallite size of Bi_2O_3 calculated from the Debye-Scherrer equation at 300°C is close to that one obtained through X'pert HighScore Plus.

4)Scanning electron microscope (SEM)

The material is analyzed using an SEM scanning electron microscope and this technic fixates on the ray of elevated-energy electrons to simulate multiple waves at the exterior of solid samples, the signals that emanate from electron-sample exchanges unveil information regarding the sample including superficial morphology and chemicalarrangement, and crystalline morphology and positioning of materials assembling the sample.**[6],** It is realized in apparatus type QUANTINA 650 FEI.

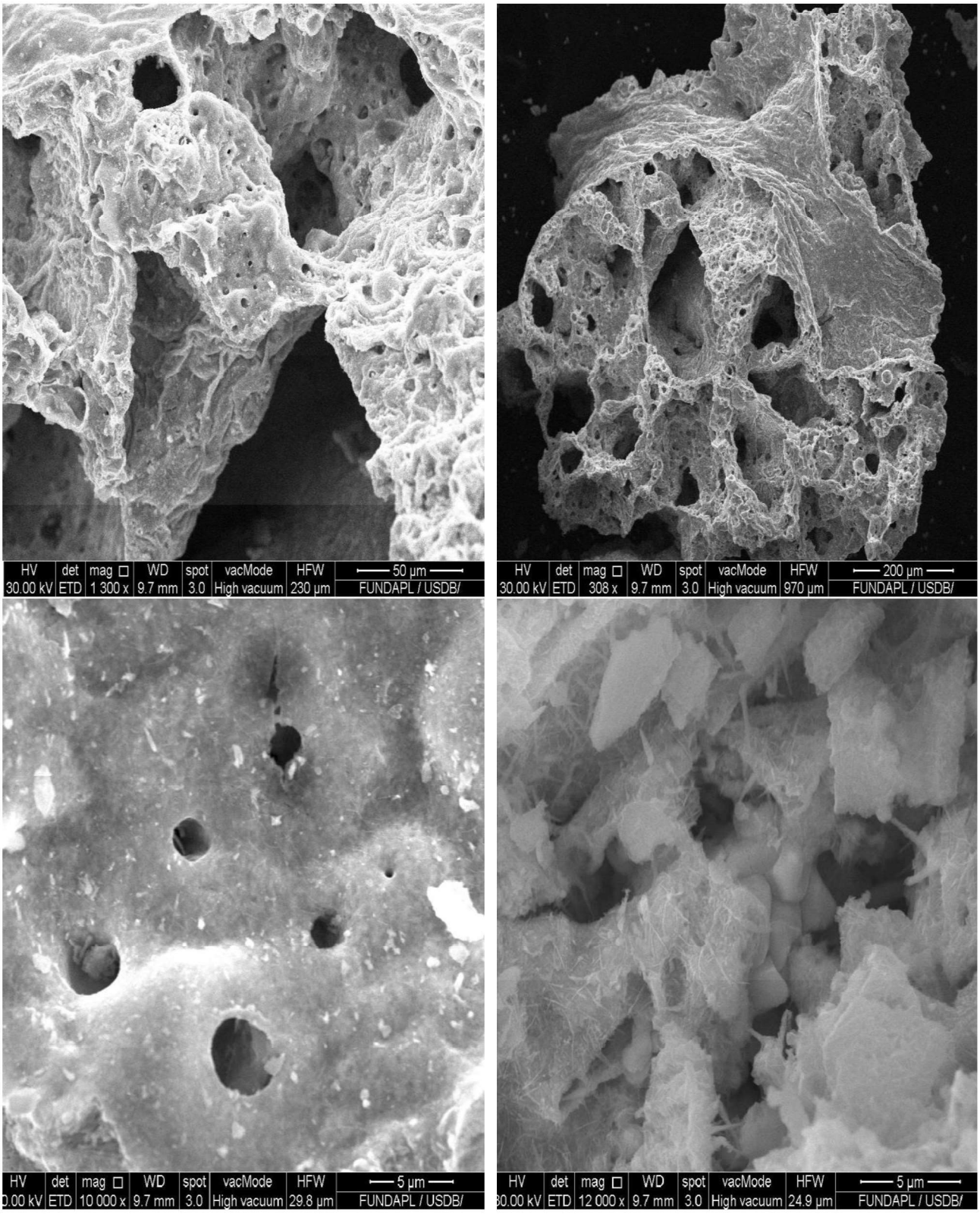


Figure 4: SEM images of the material

The SEM analysis (Fig. 4) shows the morphology of Bi-Fe-O material. It exhibits a porous surface with a high porosity, due to the NO_x and CO_2 gas release during calcination step of the gel. Such result was previously observed in a conventional sol gel preparation **[7]**. Micrographs reveal also agglomerated particles with with heterogeneous particle size (5µm to 20 µm).

5)Energy dispersive x-ray spectroscopy (EDX)

Energy-dispersive X-ray spectroscopy (EDX or EDS) is an analysis procedure role is to inspect the composition of the materials that's only in solid once **[8].**

The material was analyzed by EDX apparatus the type detected is XFLASH 6I10 BRUKER.

The EDX analysis (Figure. 5) validates the presence of Bi, Fe and O without any other polluting element, indicating the purity of the prepared material.

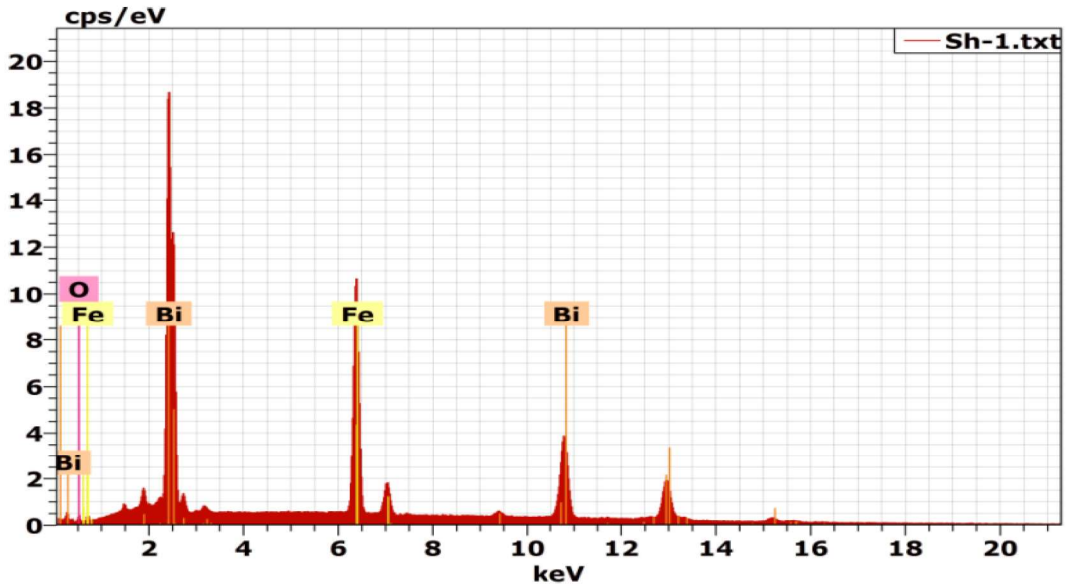


Figure 5: EDX spectrum

The percentages of each element is close to the nominal composition (Table 3) and the major part of the surface is covered with bismuth and iron more than oxygen.

Table 3: Percentage of Bi, Fe and O

Elements	Theoretical %	Experimental %	Δ%
Iron	25,08	17,85	7,23
Bismuth	66,80	60,11	6,69
Oxygen	15,34	0.89	14,45

Table 3: Theoretical and experimental percentage in EDX

6)X-ray fluorescence (XRF)

X-ray Fluorescence (XRF) its an analytical procedure that employs the interaction of X- rays with a material to showcase its elemental composition, XRF is appropriate for every form and there are two principal XRF processes **[9]**:

- energy dispersive (EDXRF).
- wavelength dispersive (WDXRF).

The analysis XRF was performed on PRIMUS II Rigaku apparatus.

Table 4 illustrates the nominal content of each element.

Elements	Theoretical %	Experimental %	Δ%
Iron	25,08	22,04	3,04
Bismuth	66,80	68,52	2,28
Oxygen	15,34	-	-

Table 4: Theoretical and experimental percentage in XRF

The nominal contents obtained for both Bi and Fe derivate slightly from the theoretical ones (3.04 and 2.28) respectively. This small difference could be due to the experimental weight loss during the preparation step of the material.

Reference

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General Conclusion

General conclusion

The objective of this framework is the preparation via sol-gel method of a mixed oxide Bi-Fe-O calcined at 300°C for 5 hours in the purpose to be studied ultimately in photocatalytic reaction for hydrogen production .

The mixed oxide Bi-Fe-O was characterized by different techniques: Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), X-ray diffraction (XRD) , scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDX), X-ray fluorescence (XRF).

Some conclusions were gathered :

- The Fourier transform infrared spectroscopy spectrum show stretching vibration corresponding to metal-oxygen bond M-O-M, indicating mixed oxide formation.
- The thermogravimetric analysis of Bi-Fe-O gel presents four weight losses up to 400°C . the formation of Bi-Fe-O oxide starts at 400°C.
- The spectrum of XRD reveal that $BiFeO_3$ prepared by sol-gel method is not obtained at calcination temperature of 300°C.
- The median crystallite size calculated from Debye-Scherrer is around 28 nm.
- The imagery obtained by scanning electron microscope display irregular and porous morphology with heterogeneous particle size between in the 5-20 µm range .
- Energy dispersive x-ray spectroscopy analysis confirms the relative abundance of mixed oxide elements of both bismuth and iron covering the surface .
- X-ray florescence elements percentages are relatively close to the theoretical ones.

Perspective

In the work presented , the obtained materials Bi_2O_3 and $Bi_{24}Fe_2O_{39}$ will be analyzed for electric properties and will be tested for photocatalytic activity and for the ideal perovskite of general formula $BiFeO_3$was not obtained by sol-gel method at a calcination temperature of 300°C.
Therefore we plan to further improve the operating conditions to obtain the pure phase which will be tested in a photocatalytic reaction for hydrogen production.