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(57) Abstract:

ABSTRACT A PROCESS FOR PREPARATION OF YTTRIUM ALUMINUM BORATE BASED FUNCTIONAL NANOMATERIAL FOR LIGHT EMITTING DIODE The Present disclosure relates to an economical method for preparation of highly efficient Yttrium Aluminum Borate (YAB) nanomaterial sample which showed photoluminescence properties in the entire range of ultraviolet as well as Bluish white colored LED at 400-451 nm wavelength in the Visible region having high coloring index. The disclosed method yields nanomaterial with characteristic band gap energy and can be used in numerous application without any hazard to human or animals from thus prepared LEDs.

F O R M 2

THE PATENT ACT, 1970

(39 of 1970)

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THE PATENTS RULES, 2003

COMPLETE SPECIFICATION

(See section 10 and rule 13)

Title of Invention:

"A PROCESS FOR PREPARATION OF YTTRIUM ALUMINUM BORATE BASED FUNCTIONAL NANOMATERIAL FOR LIGHT EMITTING DIODE"

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The following specification particularly describes the invention and the manner in which it is to be performed:

FIELD OF INVENTION

The present invention relates to an economical process for preparation of Yttrium Aluminum Borate (YAB) nanomaterial, with characteristic photoluminescence in the entire UV region as well as visible region with high coloring index for applications in light emitting diodes.

BACKGROUND OF INVENTION

In Recent time Light Emitting Diodes (LEDs) have gained significant market interest for wider array of application ranging from mobile phone application, street lights and even in water purification.

LEDs are semiconductor diodes that emit light when an electrical current is applied in the forward direction of the device. The electrical voltage is applied large enough for the electrons to move across the depletion region and combine with a hole on the other side to create an electron-hole pair. Due to this, the electron releases its energy in the form of light, and the result is an emitted photon.

LED can emit light in UV Region (100 nm to 400 nm), Visible region (400 nm to 700 nm) and IR regions (above 700nm) that depends upon the properties of the chemical compound. If any particular chemical compound shows photo luminous property in UV region and emits light.

Some of the literature reference and their disclosure are discussed herein as below:

Yoshida et.al, (2006), disclosed the luminescence properties of $YAl_3(BO_3)_4$ and $Y_{1-x}Sc_xAl_3(BO_3)_4$ under vacuum ultraviolet (VUV) light excitation at low temperatures. They also observed the change of lattice parameters of YAB while doping Sc^{3+} ions. Lattice parameters are increased when increasing the Sc^{3+} concentration. Two emission bands peaking at 3.2 and 3.9 eV are observed in pure YAB.

Zhang et.al, (2007),disclosed white light emission in $GdAl_3(BO_3)_4:Dy^{3+},Ce^{3+}$ phosphor nanorods by combustion technique at 950°C for 15 hours. It was found that the nanorods have average diameter value of 60 nm and the nanorods had emissions at 480 nm,

575 nm, and 665 nm when excited by both ultraviolet-blue and infrared light. The $GdAl_3(BO_3)_4:Dy^{3+}$ phosphor gave optimum yellow emission at 3 mol% and Ce^{3+} had an optimum value of 7 mol%.

Li & Wang (2007),disclosed the photoluminescence properties of $(Y,Gd)Al_3(BO_3)_4:Tb^{3+}$ phosphor under VUV excitation at 147 nm and found that the phosphor had green emission center at about 541 nm which is due to the electronic transition.

Ren et al (2008), disclosed synthesis of $YAl_3(BO_3)_4$: Tb^{3+} and found an emission spectrum comprised of four groups of emission lines located at about 485 nm, 541 nm, 590 nm, and 622 nm under 237 nm excitation.

Dotsenko et al (2010), discloses the luminescence properties of Sm^{3+} ions in $YAl_3B_4O_{12}$ and found that the results of electronic structure calculations of YAl3B4O12, they found that optical absorption edge (E_{edge} = 7.3 eV).

There are many chemical compound which are used to show this light emitting property in UV Range namely GaAs (Gallium Arsenide), GaN (Gallium Nitride), AlGaN (Aluminum Gallium Nitride), ZNO (Zinc Oxide) etc. Currently existing UV LED are largely made from Gallium nitride/aluminum gallium nitride (GaN/AlGaN) which takes nearly 3.5 V power and give 100Mw power output and shows this property at specific wavelength of 365 nm wavelength which is Deep Blue UV Range. Such limited / specific display of properties at particular wavelength limits its application at other wavelengths. Further the cost factor makes it less desirable for many other viable applications.

Further in the visible range, the present commercial Light emitting diodes (LED) are generally made of Indium Gallium Nitride (InGaN) chip which emits blue light. This blue light is partially converted to yellow by a Cerium-doped Yttrium Aluminum Garnet (Ce3+: YAG) phosphor placed just above the LED emitter. After combining blue light from the LED with yellow light from a phosphor powder, white light is obtained but it contains a lanthanide rare-earth element. Moreover due to a lack of a red-light component, these phosphors produce bluish white emissions with low values of their “Color Rendering Index”. Ultimately this yields optically-uncomfortable light-sources which may result retinal damage and may disturb the biological clock of healthy human being, animal and plant kingdom.

To further aggravate the shortcomings of such LEDs due to their sparse distribution in the earth's crust and low aqueous solubility, the lanthanides have a low availability in the biosphere. The prices of lanthanides are continuously increasing. Therefore, the use of rare earth element should be avoided. Also, to improve the overall photoluminescence efficiency, the absorption of the excitation light should be increased.

Therefore although there is a lot of research going on for LED development, the existing state of the art depicts that there is a strong need for a low cost material, which has a wider scope of application, is easy to prepare and is able to overcome shortcomings of prior art such as availability of starting material and cost factor, and its wider application in both UV and visible region.

SUMMARY OF THE INVENTION

The present invention explores the utilization of Yttrium Aluminum Borate (YAB) for LED applications, offering a cost-effective and efficient alternative to traditional materials. Through extensive research, the inventors identified YAB as a promising candidate due to its abundance in the earth's crust and its favorable optical properties, particularly in the UV region. Experimental findings revealed that YAB emits bluish-white light within the visible spectrum, making it suitable for LED technology.

The method disclosed in the invention facilitates the production of YAB-based nanomaterials with enhanced photoluminescence properties across a broader range of wavelengths. Specifically, the nanomaterial exhibits photoluminescence in the entire UV region (100 nm to 400 nm) and emits bluish-white light within the visible range (400 nm to 500 nm) with a high coloring index. This nanomaterial, characterized by a band gap energy of 1.343 eV, holds potential for various applications such as disinfection, water purification, and LED lighting for mobile devices, homes, and streets.

Moreover, the invention addresses safety concerns associated with traditional LED materials by offering a user-friendly alternative that poses minimal risk to the naked eye. The preparation method, employing a cost-effective sol-gel approach, ensures the efficient

synthesis of YAB nanomaterials, which undergo rigorous characterization through analytic techniques including TGA-DTA, X-ray Diffraction, FTIR Spectroscopy, and SEM analysis.

One of the key advantages of the disclosed YAB nanomaterial is its versatility in emitting light at different wavelengths, ranging from 240 nm to 400 nm, covering various levels of ultraviolet rays (UV-A, UV-B, and UV-C). Additionally, the nanomaterial demonstrates higher efficiency, requiring only 1.33 volts of power compared to traditional compounds. Its characteristic direct and indirect band gaps (1.748 eV and 1.33-1.28 eV, respectively) further contribute to its energy-efficient nature, making it superior to conventional UV LED materials like GaN/AlGaN in terms of energy consumption.

Overall, the invention presents a significant advancement in LED technology, offering a commercially viable solution for UV and visible LED lighting with improved safety, efficiency, and cost-effectiveness. By leveraging the unique properties of YAB nanomaterials, this innovation addresses key requirements such as high coloring index, low power consumption, and avoidance of lanthanide and rare earth elements, thereby paving the way for the development of next-generation LED devices.

BRIEF DESCRIPTION OF THE DRAWING:

The invention will be better understood when consideration is given to the annexed drawings wherein:

FIG. **1 (a-c)** illustrates XRD pattern of YAB annealed at different temperatures thermal Analysis.

FIG. **2** illustrates TGA-DTA curve of YAB to analyze the phase of the prepared material.

FIG. **3(a)** depicts PL Spectra of YAB at different annealing temperature. FIG. **3(b)** shows PL Spectra in UV range.

FIG. **4** depicts V-I characteristic curves of yttrium aluminum borate annealed at 1000^{o}C for 4 hours.

DESCRIPTION OF THE INVENTION

Various exemplary embodiments of the present disclosure are described herein below to enable a person of ordinary skill in the art to make and use the present disclosure.

The terminology used herein is for describing particular /embodiments only and is not intended to limit the invention.

As used herein, the singular forms "a," "an," and "the" are intended to include the plural forms as well, unless the context clearly indicates otherwise.

It will be further understood that the terms "comprises," and/or "comprising," and/or "including," when used in this specification, specify the presence of stated features, integers, steps, operations, elements, and/or components, but do not preclude or rule out the presence or addition of one or more other features, integers, steps, operations, elements, components, and/or groups thereof.

While this description has disclosed certain specific exemplary for illustrative purposes, various modifications will be apparent to those skilled in the art which do not constitute departures from the spirit and scope of the invention as defined in the following claims, and it is to be distinctly understood that the preceding descriptive matter is to be interpreted merely as illustrative of the invention and not as a limitation.

The inventors in the present invention while researching various solution to the problems identified in the background came across various approaches to find out suitable material for LED application, and during research the inventors surprisingly found that Yttrium Aluminum Borate (YAB) can be explored for its application in LEDs, YABs can be can be found on the earth’s crust in abundance and it is not Lanthanide. Further it has high coloring index and have shown their presence in UV Region.

The inventors during experiment found UV-VIS-VIS Spectrometer shows that it emits blue light (bluish white light) in the visible region.

The present invention provides a cost-effective method for yttrium aluminium borate (YAB)based low-powdered cost-effective functional nanomaterial for light emitting diodes.

The method according to present invention leads to a yttrium aluminium borate nanomaterial with improved photoluminescence property in a wider range of wavelength.

In some exemplary embodiment the method provides Yttrium Aluminum Borate (YAB) nanomaterial characterized by a photoluminescence in the entire ultraviolet (UV) region of 100 nm to 400 nm wavelength and a bluish white colored LED wavelength ranging from of about 400 nm to about 500 nm, preferably about 400 nm to about 451 nm wavelength in the visible range having high coloring index.

In one embodiment according to present invention the nanomaterial prepared in the presently disclosed method is a low-cost and low-powered nanomaterial having band gap energy 1.343 eV and can be used for various applications such as a disinfectant, water purifier, UV curing purpose, lithography, to Mobile phone backlight, PDA backlight, home LED light, street light.

In one aspect the nanomaterial prepared according to present invention can be used in LEDs which are user friendly and do not put stress or cause any harm to the naked eye.

In some exemplary embodiment according to the present invention the said nanomaterial is prepared by cost-effective chemical-based sol-gel method and analyzed by analytic techniques.

In some aspect of the above embodiment the phase analysis of the nanomaterial prepared according to present invention method is done using TGA-DTA.

In one aspect of the above embodiment the luminescent properties of the nanomaterial were investigated using Photoluminescence.

In another aspect of the above embodiment the phase identification of the nanomaterial was done using X-ray Diffraction (XRD).

In yet another aspect of the above embodiment the nanomaterial was characterized by Fourier Transform Infra-Red (FTIR)Spectroscopy to analyze functional groups and bond stretching.

In some aspect of the above embodiment the nanomaterial prepared we investigated for morphology measurement using Scanning Electron Microscope (SEM); the optical properties and corresponding band gap calculation were evaluated using UV-VIS-NIR spectroscopy.

In one key embodiment the present invention provides Yttrium Aluminum Borate (YAB) nanomaterial which shows its light emitting property at different wavelength. Non-limiting wavelength at which the nanomaterial shows light emitting property is 240 nm, 280 nm, 320 nm, 360 nm and 400 nm respectively. In some aspect according to above embodiment the emitting property at wider wavelength promotes the application of prepared nanomaterial in very wide range from all levels of ultraviolet rays i.e. UV-A, UV-B and UV-C which is characteristic feature.

In one exemplary embodiment according to present invention the method disclosed herein provides Yttrium Aluminum Borate (YAB) nanomaterial, which is of higher efficiency and characteristically takes only 1.33-volt power which is far less than 3.5 volt in case of other traditional chemical compound.

In another embodiment according to present invention the method disclosed herein provides Yttrium Aluminum Borate (YAB) nanomaterial with a characteristic direct band-gap of the YAB at about 1.748 eV.

In yet another embodiment according to present invention the method disclosed herein provides Yttrium Aluminum Borate (YAB) nanomaterial with a characteristic indirect band-gap calculated to (1.33- 1.28) eV which is equivalent to GaAs but much lesser than gallium nitride/aluminum gallium nitride (GaN/AlGaN) i.e. (3.4 to 6.2 eV) used normally for the production of UV LED. Therefore, energy consumption for UV LED prepared with Yttrium Aluminum Borate (YAB) nanomaterial of present invention method will be very less in comparison to all traditional UVLED.

It some aspect according to present invention, the band gap of YAB nanomaterial as prepared in the disclosed method is same in all regions namely UV, Visible or IR. This confirms characteristic property which will be very useful for the production of LED in Visible region as well as IR Region using the prepared nanomaterial.

Therefore, the present invention provides nanomaterial for commercial LED in UV region and Visible LED light which possess following features, such as:

- It is prepared from a general element/compound, thereby avoids use of lanthanide and rare earth element due to their negative side effects which are evident in prior art. For example side effects such as it can damage retina of the eye and can also affect biological clock of the body. Such side effects are avoided by use of prepared nanomaterials.
- It should be of High coloring Index.
- It should require less power.

In one exemplary embodiment the preparation process according to present invention for characteristically efficient Yttrium Aluminum Borate (YAB) nanomaterial comprises a SOL GEL method for Light Emitting Diode (LED) Application.

In yet another exemplary embodiment the said YAB nanomaterial preparation process uses a combinations of YAB (Yttrium Aluminum Borate) in the proportionate ratio (stoichiometric Ratio) of Y:AL:B::1:3:4 .

EXAMPLES:

Some embodiments of the present invention are exemplified below with reference to the accompanying figures in the specification:

<u>Example 1</u>: **Preparation process of Yttrium Aluminium Borate (YAB) nanomaterial sample:**

The prepared YAB nanomaterial comprises a combination of YAB (Yttrium Aluminum Borate) in the proportionate ratio (stoichiometric Ratio) of Y:AL:B::1:3:4, for which 3.4698g/mole of Yttrium Nitrate, 11.2539 g/mole of aluminum Nitrate and 2.4732g/mole of boric acid and 5.7636g/mole of citric acid were taken.

<u>Methods</u>:

Step 1:

A precursor in the form of an aqueous solution of 3.4698g/mole Yttrium Nitrate, 11.2539 g/mole Aluminum Nitrate and 5.7636g/mole Citric Acid were mixed in the deionized water. In the second beaker, 2.4732g/mole Boric Acid was dissolved in water. Then the two solutions of beaker were stirred for some time to make it complete liquid. Then both beaker solutions were mixed and placed under reflux for 2 hours on magnetic heating untill a resulting GEL is formed.

Step 2:

Then the step 1 gel was kept under microwave oven at 90^{o}C for 12 hours to evaporate all water content from gel and achieve an amorphous solid powder. Then the solid powder was crushed in mortar pistol, yielding around 7gm of powder.

Step 3:

From the step 2 powder sample about 1.2 gm of a first sample was kept under heating treatment in muffle furnace for annealing at 600 degree for two hours, the sample turned black after heating treatment. The sample is called annealed sample.

Characterization:

The prepared nanomaterial sample was characterized and analyzed with various tools namely Photoluminescence, UV-VIS-IR Spectroscopy, FTIR, VSM, V-I Characteristics, and coloring index. Reference to same can be found in Figure 1-3.

Figure 3(b) showed a PL Spectra of prepared sample in UV range.

- This UVC LED (100 nm - 280 nm) can be used for water purification, disinfectant, sterilization of food and medical equipment.
- UV B LED (280 nm – 315 nm) can be used for plant lighting, UV Curing, UV A (315-400nm) can be used for security (Bank notes), lithography, medical (Bilirubin and blood gas analysis. It can be used for mobile phone backlight, backlight for PDA/Games etc.

Observation:

- Step 2 samples when annealed at different temperatures starting from 600^{o}C for 2 hours and 4 hours, 700^{o}C (2 hrs & 4 hrs), 800^{o}C (2 hrs & 4 hrs), 900^{o}C (2 hrs & 4 hrs), 1000 ^{o}C 4 hrs, 1100^{o}C 4 hour. It was found that below 800^{o}C temperature, sample was not in phase.
- It was amorphous.
- On 800^{o}C and above temperature, sample was having crystal structure. XRD showed average crystal size 36 nm to 48 nm.
- Optical property showed that it can have light emitting property above 900 degrees to 1100 degree if annealed for 4 hrs.
- Synthesized YAB Nanomaterial between 900^{o}C to 1100 ^{o}C temperature for 4 hrs, the phase analysis were confirmed using thermal analysis and is shown in Fig. 2, has unique property of emitting light at sharp peak at 405 nm, 435 nm, 451 nm. It confirms emitting bluish white light in the visible region.
- The band gap energy is between 1.34 eV to 1.743 eV which is similar to GaAs and very less than AlGaN.
- Also V-I characteristic showed that it can be used for electrical conduction.

From the characterization data of the samples prepared in above example 1, it was found that the nanomaterial sample yielded in the process depending upon different annealing temperature can be used as material for:

a) A new ultra violet Light Emitting Diode (UV LED) can be developed from the process in all the wavelength ranges of UVA (315 nm to 400 nm), UVB (280 nm to 315 nm), and UVC (100 nm to 280nm). This nanomaterial can be characteristically which can be used for entire range of UV wavelength and will be having wide range of application.

b) A new Bluish White Light Emitting Diode (LED) can be developed from the process, such process having no Lanthanide and a high coloring Index will have some advantages such as it won’t affect eye if anyone sees on LED.

Further the LED as provided above, viz., a UVLED can be developed with the lowest ever power having bang gap of 1.34ev. The visible LED can also be developed with the same bang gap energy 1.33-1.28ev.

When compared with existing LEDs such as conventional UV LED works at around 3.5 volt and Visible LED works on 1.3 Volt but the present invention prepared YAB LED can work in both UV region as well as Visible region and will require power only about 1.32 volt. This is a significant technical and economic advantage.

Example 2: **Performance evaluation of Yttrium Aluminium Borate (YAB) nanomaterial sample prepared at 1000^{o}C:**

The performance of the cell, evaluated by V-I curves, was plotted by varying the load from infinite to zero. The polarization curve can be divided into three regions AB, BC and CD, which typically represent activation loss (energy required to overcome the energy barrier for the initiation of electrochemical reaction), ohmic loss (due to the resistance imposed by the porous material to the flow of ions) and concentration loss (due to low availability of ions in high current density regions), respectively. The cell shows that voltage obtained in about 0.8 volt and corresponding current is 0.8 mA. The details are depicted in Fig. 4.

WE CLAIM:

1. A process for preparing Yttrium Aluminum Borate (YAB) nanomaterial for light emitting diode (LED) application, the process comprising the steps of:

    a) Preparing a sol-gel precursor solution comprising Yttrium, Aluminum and Boron in a stoichiometric molar ratio of Y:AL:B::1:3:4;

    b) forming a gel from the precursor solution and drying the gel to obtain an amorphous Yttrium Aluminium Borate (YAB) precursor powder;

    c) subjecting the amorphous YAB precursor powder to a high-temperature annealing treatment at a temperature of ≥ 900 °C for a duration of at least 4 hours, thereby converting the precursor powder into a crystalline YAB nanomaterial;; and

    d) obtaining the crystalline Yttrium Aluminum Borate (YAB) nanomaterial exhibiting semiconducting electrical behaviour with measured current voltage (V-I) characteristics and photoluminescence suitable for LED operation,

    wherein the extended annealing duration at said temperature is essential to induce the electrical conductivity and LED-enabling properties in the crystalline YAB nanomaterials.

2. The process as claimed in claim 1, wherein the Yttrium Aluminum Borate (YAB) nanomaterial is characterized by a direct band-gap of approximately 1.748 eV.

3. The process as claimed in claim 1, wherein the Yttrium Aluminum Borate (YAB) nanomaterial is characterized by an indirect band-gap within the range of (1.33-1.28) eV.

4. The process as claimed in claim 1, wherein the Yttrium Aluminum Borate (YAB) nanomaterial is characterized by a uniform band gap across UV, visible, and infrared (IR) regions.

5. The process as claimed in claim 1, wherein the Yttrium Aluminum Borate (YAB) nanomaterial exhibits light emitting properties at wavelengths of 240 nm, 280 nm, 320 nm, 360 nm, and 400 nm.

6. A crystalline Yttrium Aluminum Borate (YAB) nanomaterial prepared by the process as claimed in claim 1, wherein the nanomaterial:

   a) is free from rare-earth dopant elements;

   b) Exhibits photoluminescence in the entire ultraviolet (UV) region including UV-A, UV-B and UV-C wavelength and emits bluish white light in the visible range with a wavelength ranging from about 400 nm to about 500 nm;

   c) possesses a band-gap in the range of about 1.28 eV to 1.74 eV; and

   d) demonstrates semiconducting electrical behaviour with measurable current-voltage characteristics enabling low-voltage LED operation at 1.3 V,

   wherein said optical and electrical properties arise from the crystalline phase formed by annealing at a temperature of at least 900 °C for at least 4 hours.

7. The Yttrium Aluminum Borate (YAB) nanomaterial as claimed in claim 6, wherein the nanomaterial exhibits non-linear V-I characteristics indicative of semiconducting behaviour when integrated into an LED test configuration.

Dated this 14th day of March, 2024

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ABSTRACT

A PROCESS FOR PREPARATION OF YTTRIUM ALUMINUM BORATE BASED FUNCTIONAL NANOMATERIAL FOR LIGHT EMITTING DIODE

The Present disclosure relates to an economical method for preparation of highly efficient Yttrium Aluminum Borate (YAB) nanomaterial sample which showed photoluminescence properties in the entire range of ultraviolet as well as Bluish white colored LED at 400-451 nm wavelength in the Visible region having high coloring index. The disclosed method yields nanomaterial with characteristic band gap energy and can be used in numerous application without any hazard to human or animals from thus prepared LEDs.

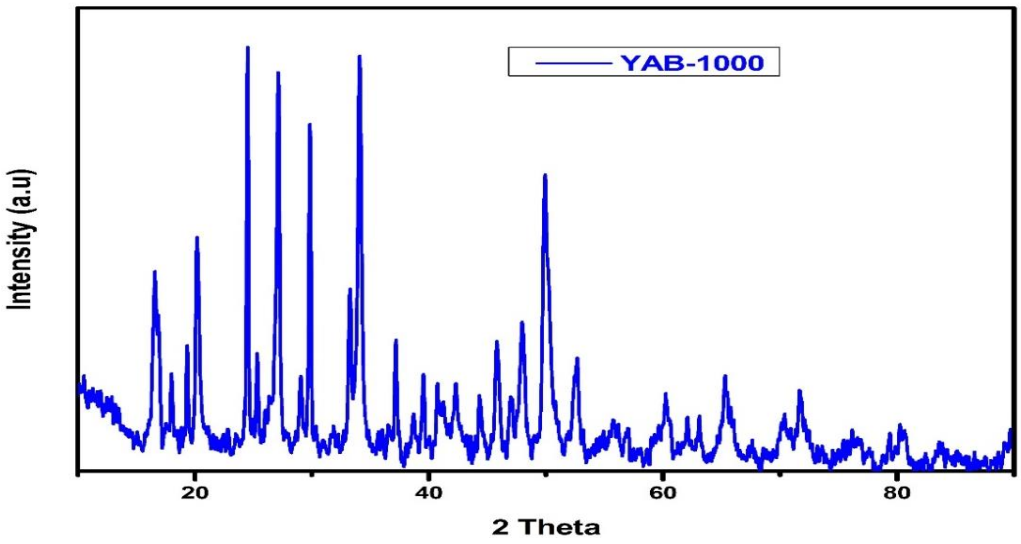


Fig. 1(a)

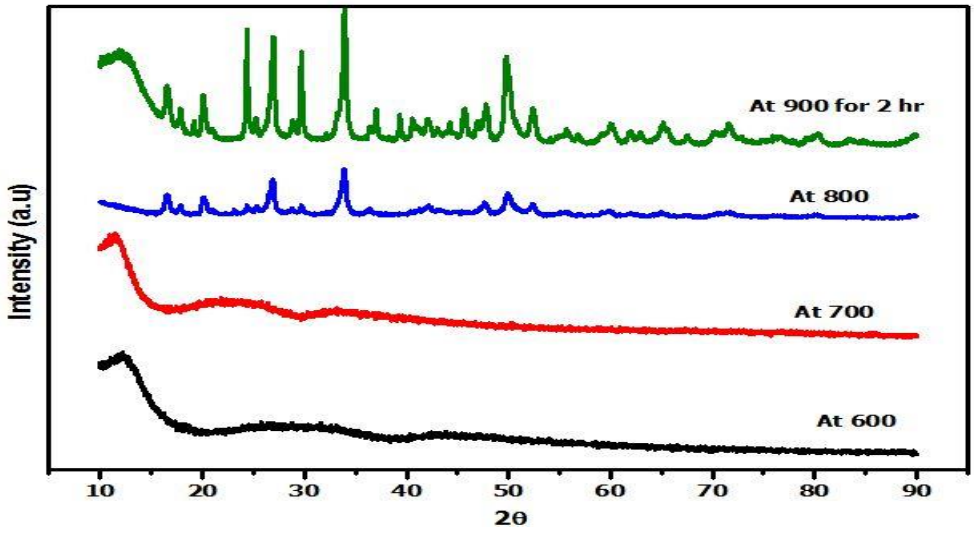


Fig. 1(b)

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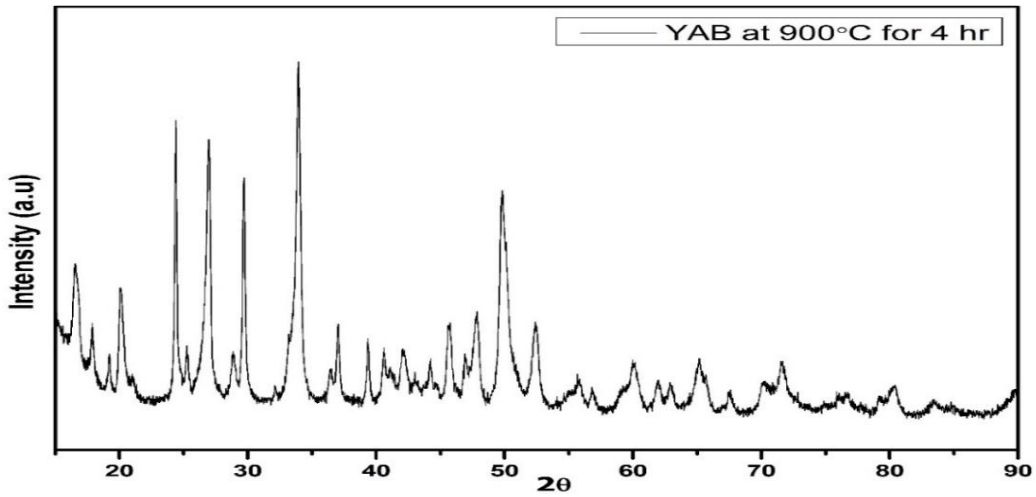


FIG. 1 (c)

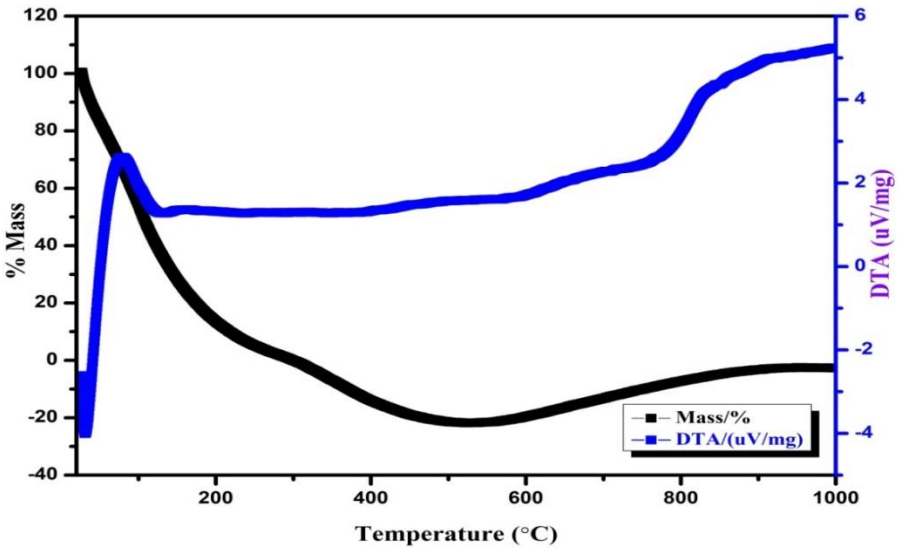


FIG. 2

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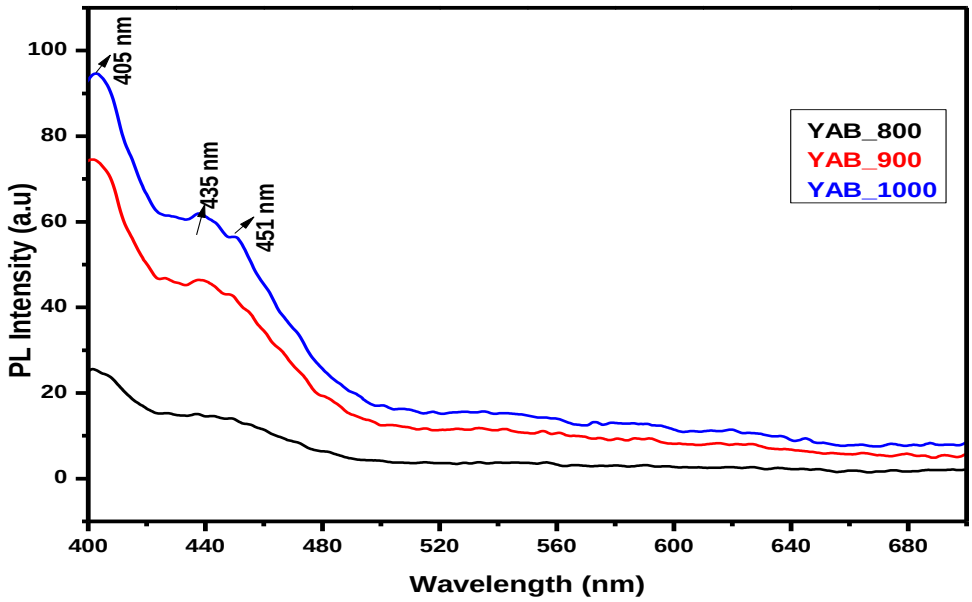


FIG. 3(a)

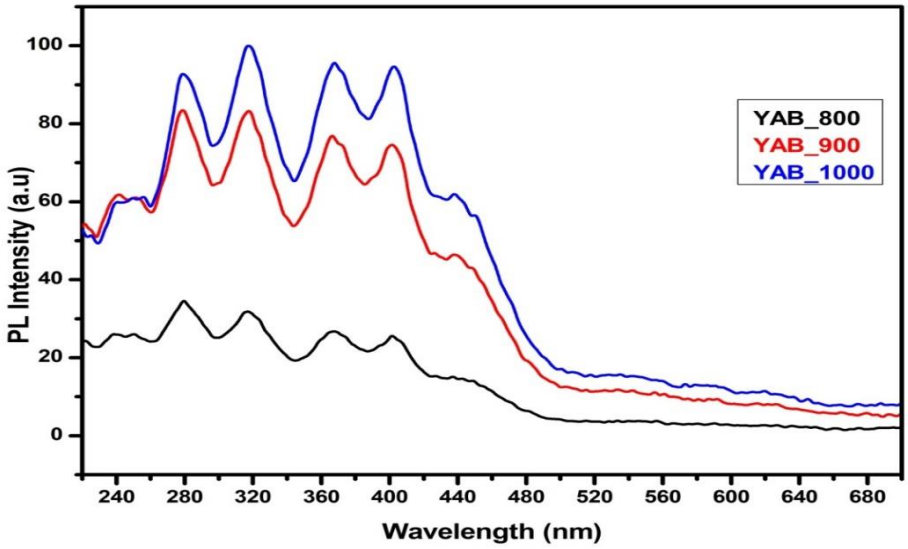


FIG. 3(b)

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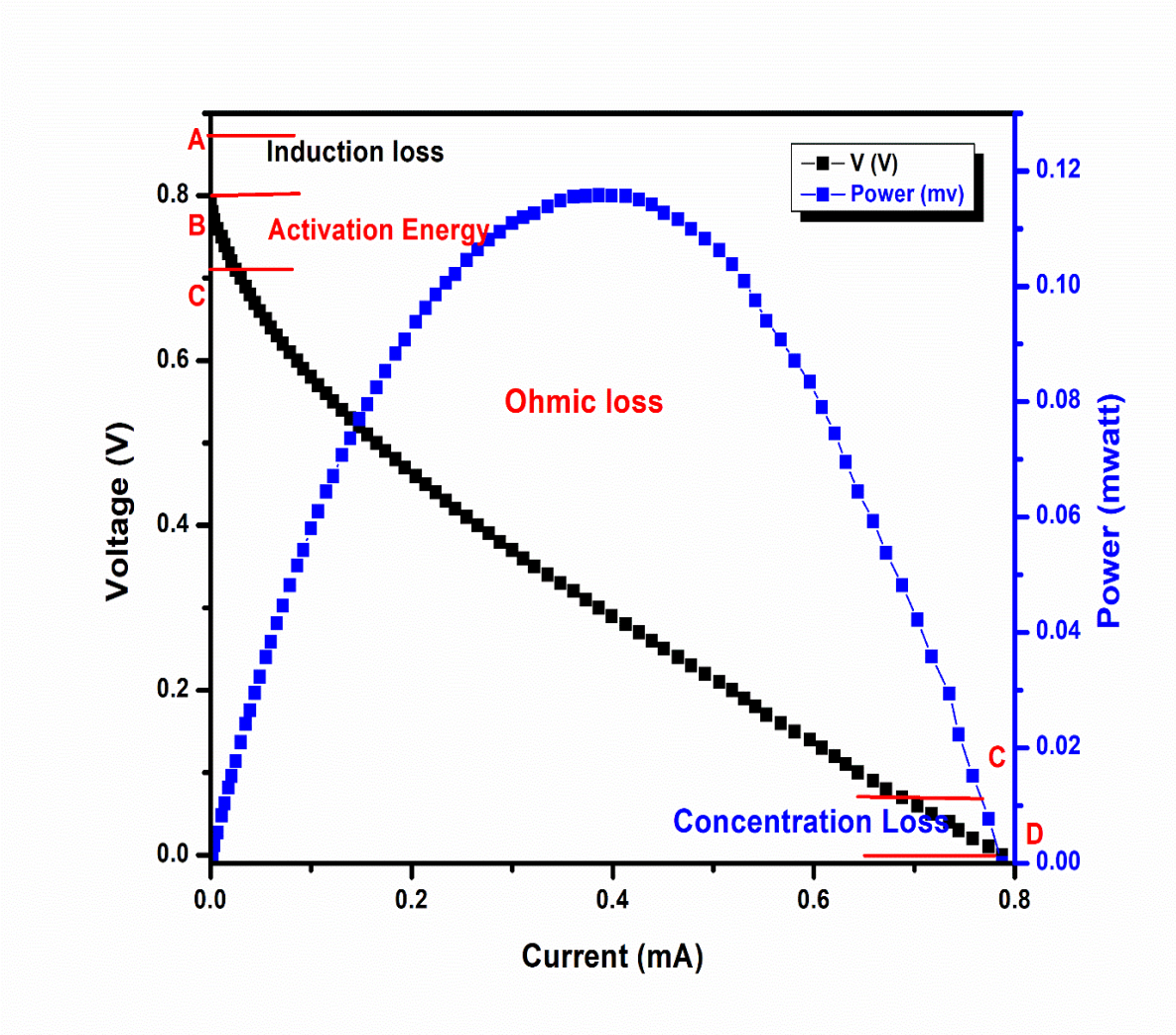


FIG. 4

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