

LIQUEFIED-PETROLEUM-GAS SENSING PERFORMANCE OF CuO–Ag₂O BIMETALLIC OXIDE NANOPARTICLES

In the present work, we have synthesized CuO–Ag₂O bimetallic oxide nanoparticles using microwave-assisted and solid state diffusion routes. The structural, morphological, optical and thermal studies of the synthesized materials were done with an X-ray diffractometer (XRD), a scanning electron microscope (SEM), Fourier transform infrared (FTIR), ultraviolet-visible (UV-VIS) and thermogravimetric analysis (TGA), respectively. Comparatively different sensing parameters such as sensing response at room temperature, operating temperature, response and recovery time and stability characteristics were investigated and discussed for liquefied petroleum gas (LPG).

NANOCRYSTALLINE STRUCTURE, MICROWAVE PROCESSING, LPG SENSING.

1. Introduction

Presently, liquefied petroleum gas (LPG) is an exciting alternative to conventional energy sources, due to its abundance and comparatively inexpensive price. This gas is odorless and colorless, and when burned, it generates less emission than petroleum. The main features of LPG include: (i) high heating value, (ii) virtual absence of sulphur, which results in clean burning and (iii) reliable quality ensuring, mainly in applications such as gas engines. Besides that LPG is highly inflammable and must therefore be stored away from sources of ignition. Moreover, LPG vapour is heavier than air, so any leakage will go down to the ground and accumulate, which is difficult to disperse.

Sivapunniam et al. [1] reported the high-performance LPG sensing materials based on zinc oxide and zinc stannate nanostructures. In order to improve the gas sensing performance of material towards LPG, surface modification of ZnO nanorods using zinc stannate microcubes was performed. A considerable improvement in sensing response was observed. The operating temperature for this material was 250 °C [1]. CuO, Al₂O₃, Ag₂O and La₂O₃ loaded SnO₂

nanoparticles were successfully synthesized by a hydrothermal route at 200 °C for 3 h in Ref. [2] for LPG sensing. Out of these prepared nanoparticles, SnO₂ sol suspension of 5 wt% doped with CuO 2 wt% showed the best sensitivity at 330 °C. Singh et al. [3] synthesized a CuO–SnO₂ nanocomposite via the sol–gel route as a LPG sensing material at room temperature. However, response and recovery times for the fabricated sensor were 180 and 200 s, which is not good practically [3]. Deore et al. [4] demonstrated the LPG sensing at considerably high operating temperature by CuO-loaded ZnO thick film fabricated using the screen printing technique.

From recently reported articles, it is observed that CuO-based sensors have high operating temperature and slow response and recovery time characteristics. Taking into account this aspect of CuO-based materials, we planned to prepare CuO–Ag₂O bimetallic oxide nanoparticles for LPG sensing at low temperature and with fast response and recovery characteristics. Various research groups across the globe reported the preparation of CuO–Ag₂O nanoparticles using different approaches. Jin et al. [5] reported

the non-enzymatic glucose sensor, which was fabricated by growing CuO–Ag₂O nanoparticles on an AgCuZn alloy substrate. These authors prepared the CuO–Ag₂O nanowires directly on the surface of an AgCuZn alloy by a facile method using the hierarchical composition of the two oxides. The nanowires synthesized in such a manner were employed for enhancing catalytic formaldehyde oxidation on CuO–Ag₂O nanowires for gas sensing and hydrogen evolution [6]. The authors of Ref. [7] described metal-metal bonding process using a mixture of CuO nanoparticles and Ag₂O nanoparticles and correlated its effect to mechanical properties. Maniecki et al. developed the bimetallic systems Au–Cu and Ag–Cu/CrAl₃O₆ as catalysts for methanol synthesis [8].

In the present work, we have synthesized CuO–Ag₂O bimetallic oxide nanoparticles using microwave-assisted and solid state diffusion routes. Different techniques were used to describe these samples. The LPG sensing characteristics were investigated at room and various other temperatures.

2. Experimental section

2.1. Microwave assisted synthesis of CuO–Ag₂O bimetallic oxide nanoparticles

The laboratory grade microwave synthesizer CEM Phoenix (Power Output is 1350 ± 50 W and Magnetron Frequency is 2455 MHz) was used for synthesis of CuO–Ag₂O bimetallic oxide nanoparticles. The starting chemicals, Cu(NO₃)₂·3H₂O and AgNO₃ were dissolved in 1 : 1 M ratio in deionized water of resistivity not less than 18.2 MΩ·cm, separately. These solutions were mixed under constant magnetic stirring for 30 min. Finally, the reaction mixture was heated in a CEM-supplied single-use vessel. The mixture-containing vessel was reacted in the CEM microwave at a temperature of 600 °C. The reaction process temperature is a crucial aspect of microwave-assisted and solid state diffusion route, which affect the particle size of the product [9].

2.2. Solid state diffusion synthesis of CuO–Ag₂O bimetallic oxide nanoparticles

CuO–Ag₂O bimetallic oxide nanoparticles were prepared by solid state diffusion routes using the starting chemicals, Cu(NO₃)₂·3H₂O

and AgNO₃. The chemicals were taken in a 1 : 1 M amount. The preparatory materials were mixed thoroughly for 2 h using an agate mortar pestle. The crushed samples were placed in a crucible and heated at 973 K in a muffle furnace for 4 h. Then they were removed, crushed in the mortar again and heated at 1173 K for 8 h. Finally, the samples were left in the furnace to cool down to room temperature.

2.3. Materials characterization

The powders were characterized structurally in an X-ray diffractometer (XRD) (Rigaku, Miniflex) with CuK_α radiation ($\lambda = 1.54$ Å), with a step size of 0.02 and a step time of 2.0 s. The morphology and size of the synthesized samples were determined by a scanning electron microscope (SEM) using a JEOL JSM-7500F, operating at 300 keV. Fourier transform infrared (FTIR) spectra were recorded on a Shimadzu FTIR spectrometer of model:8400S by the use of KBr tableting. The ultraviolet-visible (UV-VIS) spectra of samples were recorded using Agilent Cary 60. Thermogravimetric analysis (TGA) was performed on a Shimadzu DTG-60h thermal analyzer under nitrogen atmosphere.

2.4. Fabrication of sensors and gas sensing measurements

For measuring the LPG sensing response, dried powder of CuO–Ag₂O bimetallic oxide nanoparticles was dispersed with a temporary binder prepared via a previously reported method [10]. For good adhesion to a substrate, the ratio of nanoparticles to binder was kept at 90 : 10 in formulating the paste. A thick film of the synthesized nanoparticles was deposited on a chemically cleaned glass substrate of 75 × 25 mm size by using screen-printing. The resulting films were dried at room temperature (303 K) for 12 h. Further heat treatment was applied to the film at 373 K for 5 h for evaporation of volatile organic compounds from the binder. The thickness of the sensing surface was measured using a Digimatic (Japan) outside micrometer (series 293) with a resolution of ± 0.001 mm, which was found to be 12 and 17 μm for microwave-assisted and solid state diffusion routes, respectively. Highly conducting silver paste was used to make ohmic contacts on adjacent sides of the films



with an electrode thickness of 8 μm for surface resistance measurements. Both films were subjected to heating at 353 K for 15 min to dry the silver paint in an argon environment.

The electrode separation for both sensors was kept of the order of 23 mm, to avoid capacitive effect. To determine the gas-sensing performance, the sensor was loaded into a gas-sensing chamber. Important physical parameters such as temperature and humidity inside the chamber were precisely controlled. The sensing response was evaluated by using air as a background gas. The gas-sensing response (S) is defined as

$$S = [|R_a - R_g|/R_a],$$

where R_a is the resistance in air (baseline resistance), and R_g represents the resistance in gas.

The resistance of the sensors was analyzed using the voltage drop method adopted by Waghuley et al. [10]. The main parameters of the sensors were analyzed for different concentrations (ppm) and temperatures. The gas concentration within the chamber of 5 L was retained by injecting known volume of LPG using a gas-injecting syringe technique.

3. Results and discussion

In Fig. 1, the XRD pattern of CuO–Ag₂O bimetallic oxide nanoparticles exhibits broad peaks indicating nanometric dimensions of the synthesized materials. The symbolic

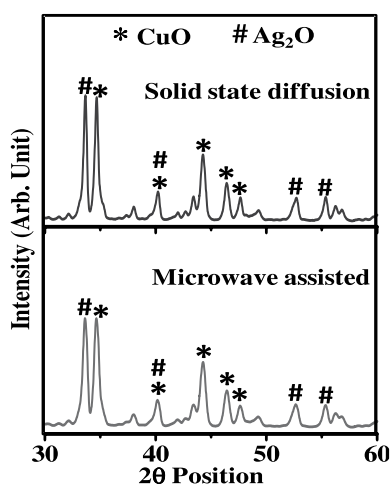


Fig. 1. XRD pattern of the synthesized CuO/Ag₂O bimetallic nanoparticles

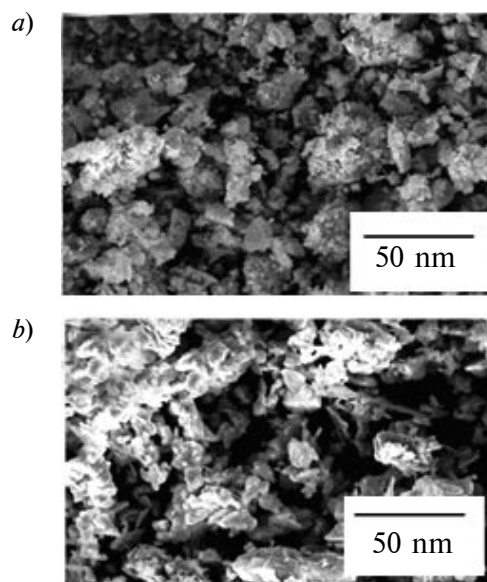


Fig. 2. SEM images of the CuO/Ag₂O bimetallic nanoparticles synthesized by Microwave assisted (a) and Solid state diffusion (b) routes

representation of diffraction peaks for CuO and Ag₂O exactly indexed to JCPDS file No. 89-5897 and 75-1532, respectively. It is also well-recognized that CuO and Ag₂O alloy possesses easy phase separation, due to the radius inequality between Cu and Ag within the alloy-nanoparticles [11]. The overlapped diffraction peak appears around 41°, which might serve as some indication of alloy formation. The crystal grain size was estimated using Scherrer's equation, which was found to be 17.3 and 20.4 nm for microwave-assisted and solid state diffusion routes, respectively.

Fig. 2 shows the scanning electron microscope (SEM) images of CuO/Ag₂O bimetallic nanoparticles synthesized by microwave-assisted and solid state diffusion routes, respectively. SEM micrograph reflects the agglomeration of nanoparticles, which may be high due to reaction temperature for both routes. The crystal grain size estimated from SEM micrographs was found to range between 18 – 21 nm. This variation in particle size strengthens with XRD analysis. Fig. 2, a shows the three-dimensional aggregate morphology of the synthesized material, which is formed by a group of primary particles. Fig. 2, b depicts that primary aggregation particles form a sheet-like shape.

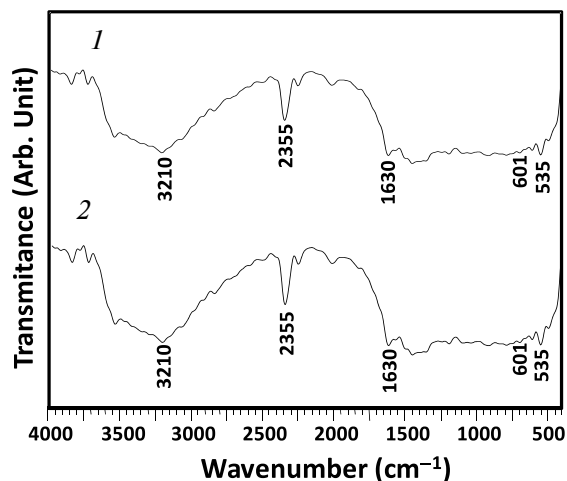


Fig. 3. FTIR spectrum of the CuO/Ag₂O bimetallic nanoparticles synthesized by Microwave-assisted (1) and Solid state diffusion (2) routes

The Fourier transform infrared (FTIR) spectra of the synthesized products are shown in Fig. 3. The FTIR spectrum of both samples match finally with each other. The double-peak at 535 and 601 cm⁻¹ is likely to be from Cu–O stretching [12]. The insignificant band at 513cm⁻¹ is assigned to Ag–O stretching vibration [13]. The broadband in the lower energy region (2200–4000 cm⁻¹) is attributed to the presence of free electron tail in inorganic materials.

In Fig. 4, we present the ultraviolet and visible (UV-VIS) optical spectra of CuO/Ag₂O bimetallic nanoparticles synthesized by microwave-assisted and solid state diffusion routes. The absorption between 300–325 nm for both routes may be assigned to a strong surface plasmon resonance in Ag nanoparticles [14]. It is also apparent from the plot that the CuO/Ag₂O bimetallic nanoparticles synthesized by the solid state diffusion route exhibit a red shift with respect to the microwave-assisted one. This is strong evidence of particle size increment [15]. This statement is also supported by XRD and SEM analyses. The results reveal that reaction route can affect the absorption properties of the synthesized CuO/Ag₂O bimetallic nanoparticles.

Fig. 5 presents the TGA curves for the CuO/Ag₂O bimetallic nanoparticles synthesized by microwave-assisted and solid state diffusion

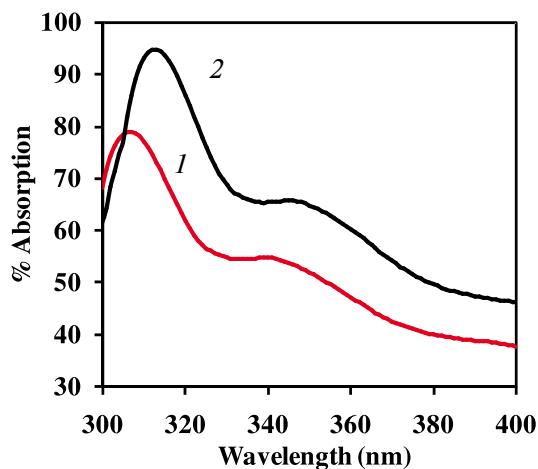


Fig. 4. UV-VIS spectra of the CuO/Ag₂O bimetallic nanoparticles synthesized by Microwave-assisted (1) and Solid state diffusion (2) routes

routes to show the thermal behavior during heat treatment. There are two major mass change steps with an increase in temperature. The weight loss up to 375 K may be attributed to the removal of constituent water molecules. The TGA curves for both samples show small weight loss in the range from 373 to 450 K. The thermal stability in this range is very important for gas-sensing materials, which is discussed later. This thermal stability was witnessed again around 600–650 K.

Fig. 6 shows the good dependence of

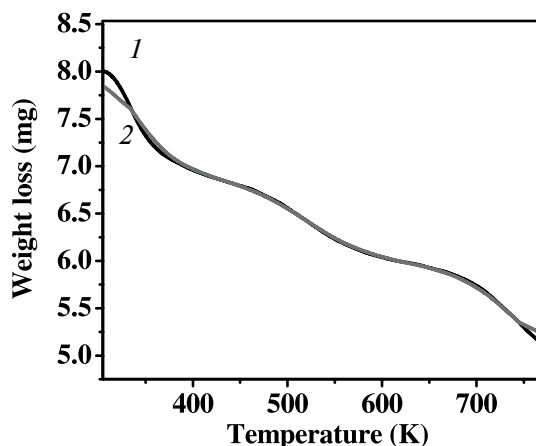


Fig. 5. Thermogravimetric traces of the CuO/Ag₂O bimetallic nanoparticles synthesized by Microwave-assisted (1) and Solid state diffusion (2) routes

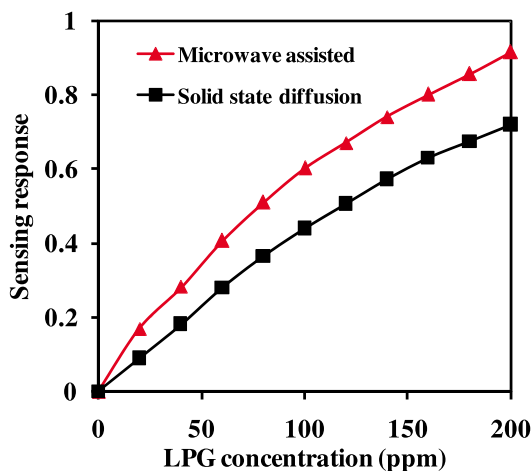


Fig. 6. LPG sensing response of the devices at room temperature

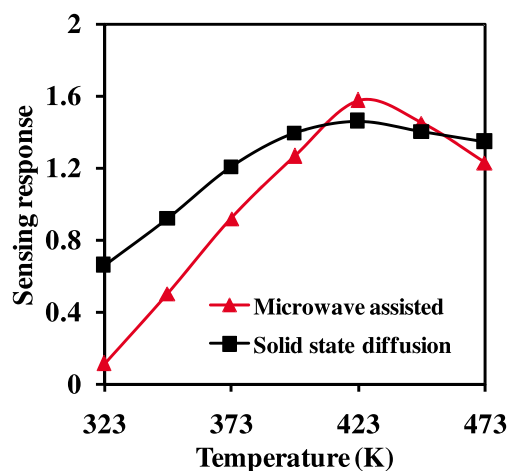


Fig. 7. Operating temperature response of sensors towards LPG

the sensing response of the CuO/Ag₂O bimetallic nanoparticle-based devices on the concentration of LPG at room temperature (303 K). The baseline resistance for sensors developed using microwave-assisted and solid state diffusion routes, were found to be 2.736 and 2.869 MΩ, respectively. Upon exposure to LPG (reducing gas) the resistance values of the both sensors increased [16]. An increase resistance of sensing surface in the presence of reducing species clearly reflects *p*-type behavior of sensing surface [17]. The sensing response of the devices was found to vary nearly linearly as a function of LPG concentration. Saturation was not observed for LPG up to 200 ppm for both sensors. This may indicate the optimum detection limit for sensors towards the LPG being beyond 200 ppm.

The response of sensors towards the 200 ppm LPG as a function of operating temperature is displayed in Fig. 7. Both curves show the highest sensing response values corresponding to 423 K, which is reported in literature as a particularly low operating temperature. This is the key accomplishment of the present investigation, which reduced the operation cost and risk of detection. The sensing response begins to decrease from a particular temperature, which may be due to desorption of atmospheric oxygen from the sensing surface [18].

Sensing response fundamentally depends on electron transfer reactions, which are redox

reactions. Gas sensing is related to oxygen vacancies which act as adsorption sites for gas molecules through atmospheric oxygen. When the sensor is exposed to LPG, it interacts with the adsorbed oxygen ions and yields H₂O and CO₂. A plausible sensing mechanism in pictographic form for LPG is shown in Fig. 8.

The transient response to 100 ppm LPG was studied for both sensors at room temperature and is displayed in Fig. 9. For this measurement, gas was inserted into the chamber and the resistance of the sensor was measured in air and in the presence of gas. Both sensors show almost the same response (16 s) and recovery (20 s) time lengths for LPG sensing.

To analyze the stability of sensors, their responses to 100 ppm LPG were measured for 30 days with an interval of 5 days at room temperature. The stability results are

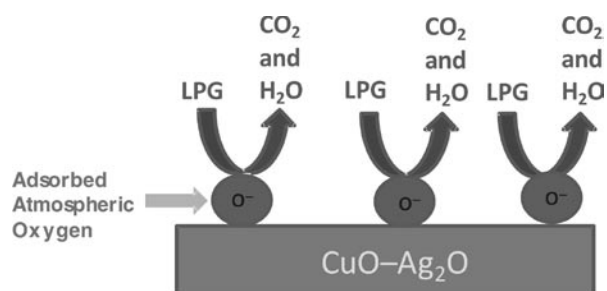


Fig. 8. Bridging oxygen having its origin in a plausible sensing mechanism for CuO/Ag₂O bimetallic nanoparticles for LPG

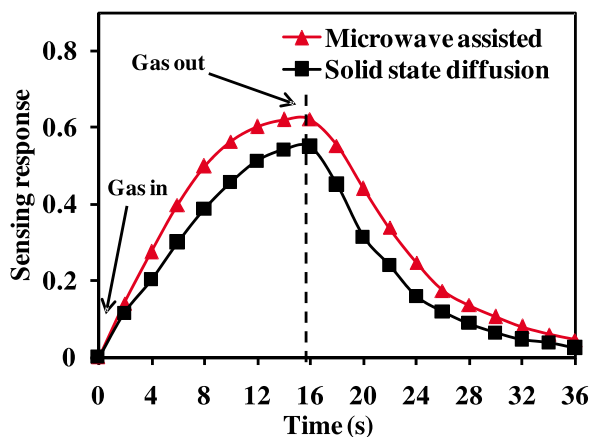


Fig. 9. Transient response characteristics of CuO/Ag₂O bimetallic nanoparticles to 100 ppm LPG at room temperature

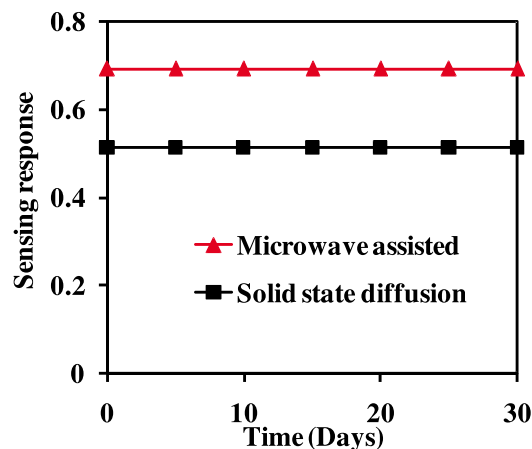


Fig. 10. Stability response characteristics of CuO/Ag₂O bimetallic nanoparticles to 100 ppm LPG at room temperature

demonstrated in Fig. 10. The sensors exhibited nearly constant response to LPG indicating the good stability.

4. Conclusions

In summary, we successfully demonstrated the synthesis of CuO–Ag₂O bimetallic oxide nanoparticles by using microwave-assisted and solid state diffusion routes. The structural conformation obtained through X-ray diffraction analysis clearly pointed out the formation of CuO–Ag₂O bimetallic oxide nanoparticles. In this comparative study, CuO–Ag₂O bimetallic oxide nanoparticles synthesized by a microwave-assisted route demonstrate good

sensing performance towards LPG, exhibiting linear sensing response at room temperature, low operating temperature, excellent stability and fast response and recovery.

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THE AUTHORS

NEMADE Kailash R.

Sant Gadge Baba Amravati University, Amravati, India
Amravati, Maharashtra 444602, India
krnemade@gmail.com

WAGHULEY Sandeep A.

Sant Gadge Baba Amravati University, Amravati, India
Amravati, Maharashtra 444602, India
sandeepwaghuley@sgbau.ac.in

Немаде К.Р., Уагхули С.А. ХАРАКТЕРИСТИКИ ДАТЧИКОВ ЧУВСТВИТЕЛЬНОСТИ К СЖИЖЕННОМУ ПРИРОДНОМУ ГАЗУ НА ОСНОВЕ МЕТАЛЛОКСИДНЫХ НАНОЧАСТИЦ CuO-Ag₂O.

Для создания датчиков чувствительности к сжиженному природному газу синтезированы биметаллические оксидные наночастицы CuO-Ag₂O путем использования СВЧ-обработки и твердотельной диффузии. Структурные, морфологические, оптические и температурные исследования синтезированных материалов были выполнены на рентгеновском дифрактометре, сканирующем электронном микроскопе, инфракрасном Фурье-спектрометре, оптическом спектрометре видимого и УФ диапазонов, а также на термогравиметрическом анализаторе. Был проведен сравнительный анализ различных параметров чувствительности к сжиженному природному газу, таких как срабатывание при комнатной температуре, рабочая температура, время отклика, время возврата в исходное состояние, стабильность. Проведено обсуждение результатов.

НАНОКРИСТАЛЛИЧЕСКАЯ СТРУКТУРА, СВЧ-ОБРАБОТКА, ДАТЧИК ЧУВСТВИТЕЛЬНОСТИ К СЖИЖЕННОМУ ПРИРОДНОМУ ГАЗУ.

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СВЕДЕНИЯ ОБ АВТОРЕ

НЕМАДЕ Кайлаш Рамбхау – *Ph.D., доцент кафедры физики университета Сант Гадж Баба Амравати, г. Амравати, Индия.*

Amravati, Maharashtra 444602, India
krnemade@gmail.com

УАГХУЛИ Сандип Анандрао – *Ph.D., доцент кафедры физики университета Сант Гадж Баба Амравати, г. Амравати, Индия.*

Amravati, Maharashtra 444602, India
sandeepwaghuley@sgbau.ac.in