Design and Development of Paper/ZnO–SnO₂ Heterostructured Ultra-Fast TENG Based LPG Sensor

Prabhabkar Yadav,¹ Ajeet Singh,² Shakti Singh,²,* and Dheeraj Kumar¹

¹Department of Electrical Engineering, Shri Ram Swarup College of Engineering and Management, Lucknow, U.P., India
²Nanomaterials and Sensors Laboratory, Department of Physics, Babasaheb Bhimrao Ambedkar University, Lucknow-226025, India

This work establishes a very simple and economical preparation of paper-based triboelectric nanogenerators for self-powered LPG sensing. Magnification in the output of TENG was achieved by ZnO/SnO₂ synthesized by hydrothermal. This material has been thoroughly characterized through XRD, UV, FESEM, FTR, and Nanozetasizer. The average crystallite size of the heterostructure was obtained as 17.59 nm. Bandgap of ZnO/SnO₂ material was found as 3.49 eV. FESEM exhibits that present heterostructure material exhibits spherical nature with lots of voids on the film surface. From Nanozetasizer, the diameter of particles resides between the range 50–80 nm with an average particle size as 63.23 nm. The fabricated TENG generates a maximum output voltage of ~75 volts which is more than the output of paper-based TENG. This TENG was used as a power source to operate a resistive LPG sensing film. The maximum response of 24 and minimum response-recovery times of 120–135 ms were observed, which makes this TENG sensing device ultra-fast.

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Nanogenerator converts extremely tiny mechanical vibrations or thermal differences into electric power. As produced by small-scale physical change which is based on piezoelectric (change in dipole), pyroelectric (change in temperature), and triboelectric (change in triboelectricity) phenomena.1–2 This type of energy harvester converts external kinetic motion into electrical power by a nanostructured material. Mainly, these are of three types: (i) Piezoelectric Nanogenerator (PENG), in which the temperature difference of the device drives the charge carrier. (ii) Piezoelectric Nanogenerator (PENG), in which the change in the dipole moment of the material through mechanical force generates the output voltage, which was firstly invented by Prof. Z.L. Wang in 2006.3 The working principle of the nanogenerator can be explained in two ways with respect to the force exerted (a) perpendicular and (b) parallel to the axis of the nanowire. (iii) Triboelectric nanogenerator (TENG),4 in which external mechanical energy converts into electricity by a fusion of the electrostatic induction and triboelectric effect. In addition, this new energy harvesting technology also incorporates a number of other properties, such as economical manufacturing and assembly, exceptional sturdiness and consistency, and ecological friendliness. The TENG can be used to produce all kinds of mechanical motions that are present or unexploited in our day-to-day life; such as body motion, vibration, revolving tire, blowing wind, flowing water, etc.5–8

Piezoelectric and triboelectric nanogenerators have enough output suitable for a self-powered system. The triboelectric nanogenerator gives a suitable output and doesn’t need rafting. Both organic and inorganic materials can be used based on their triboelectric behavior. Nanogenerators can harvest waste energy from the environment and thus are a sustainable energy source. The amount of power generated by the nanogenerator is comparatively small. Also, organic materials-based nanogenerators are limited to small duty sequences and decay very swiftly.

The limited availability of conventional energy sources is another big issue to overcome the increasing energy demands. In recent decades, researchers from the scientific community are focusing on new renewable energy tools/technologies.7,9 Piezoelectric/triboelectric nanogenerators (PENGs/TENGs) are very promising renewable sources for the conversion of mechanical energies into electricity. Various types of organic/inorganic nanomaterials are used to make smart and new types of PENGs/TENGs. Several nanomaterials are used in different types of resistive sensors and energy-based devices.10–12 ZnO is rigorously used to sense different types of gases.13–15 This research work reports the pitfalls of nanogenerators and gives a better pathway for remarkable improvement in performance. The smart PENGs/TENGs nanogenerators are auspicious explanations for producing an improved class of self-sufficient and self-powered electronic devices.

Nanogenerators are described as a field that practices displacement current as the driving energy for efficiently translating mechanical motion into electric output, discounting whether nanomaterials are used or not. A diagram of TENG is denoted in Fig. 1.16–22 It consists of a cell having an electrode and Keithley electrometer with GPIB connected computer. Triboelectrification is a well-known effect,9–13 but it is typically taken as an undesirable effect and is evaded in many technologies. A TENG is used to change mechanical power into electric power in combination with triboelectrification and electrostatic induction.24–27 In this power production unit, in the internal circuit, a potential is generated by the triboelectric phenomenon because of the charge transmission between 2 thin films (Material 1 and Material 2 in the schematic diagram) that displays reverse tribo-charge. In the superficial circuit, electrons move between two probes connected on the back sides of the tribo layer to equilibrium the potential. The spacers are used to separate the two materials from direct contact. A digital multimeter (Keithley 6517 B) was attached to the electrodes of TENG in order to measure output parameters like voltage, current, charge, power density, etc.

The TENG is applied to produce electrical power from all types of mechanical power. Alternatively, TENG is also be used as a self-driven sensor for dynamically sensing the static and dynamic processes ascending from mechanical distresses using the output power of the TENG. Although there are many types of materials are previously used as tribo layers, this work utilized used paper as one layer in TENG because of the following 3 reasons: (1) paper is biodegradable, hence there is no negative environmental impact, (2) Used paper is cost-effective because there is no purchasing cost in the used paper, and (3) paper is highly electropositive in triboelectric series, hence it easily accepts electrons from opposite layer, which enhances output.

As a new energy-generating device, TENGs are able to convert nearly all mechanical energies into electric power. However, the same preparation procedure necessitates coating with conductive materials, which restricts their real-time applications. In this work, a TENG is designed using cellulose paper/ZnO-SnO₂ nanomaterial. This TENG is used as a power source for a resistive ZnO-SnO₂ film for LPG sensing.

*E-mail: shakti665@gmail.com
Experimental

**Required materials.**—Zn(CH$_3$COO)$_2$.2H$_2$O (99.999% pure), Cellulose paper, SnCl$_4$.4H$_2$O (99.995% pure), Copper tape for electrodes, copper wires, distilled water, and NaOH (98.999% pure) are the materials required for synthesis and fabrication of desired TENG.

**ZnO/SnO$_2$ heterostructured synthesis.**—ZnO/SnO$_2$ heterostructured nanocomposite material was synthesized using a one-step hydrothermal process. For this purpose, zinc acetate, tin chloride, ethanol, and distilled water were purchased from sigma Aldrich USA. All the regents are 99% pure and used without purification. 0.2 M Zn(CH$_3$COO)$_2$.2H$_2$O with 0.1 M SnCl$_4$.4H$_2$O were mixed in 50 ml distilled water and stirred at room temperature. Further, the aqueous solution of NaOH was added slowly drop by drop to the solution with constant stirring until the neutral pH. This mixture was then moved to a 100 ml stainless-steel autoclave (Teflon-lined) and kept in the microwave oven at 160 °C for 18 h. Then cooling of the solution to room temperature was done. The found solution was washed and filtered. Later it was dried at 85 °C for 6 h in an air oven and annealed at 550 °C for 3 h @ 2 °C min$^{-1}$ in a furnace (programmable). The end product gained was ZnO/SnO$_2$ heterostructure. The whole process of synthesis of ZnO/SnO$_2$ is portrayed with the help of Fig. 2.

**Characterizations.**—XRD plot of the ZnO/SnO$_2$ heterostructure was analyzed by Bruker D8 advanced Ecosystem X-ray Diffractometer, functioning with monochromatic Cu-K$_\alpha$ as the radiation source. The morphology of the surface was investigated by FE-SEM (Jeol-JSM 7601 F). The particle size of heterostructure material was measured by Nanozetasizer-NSZ90. Band gap analysis was performed by Evolution 201 UV–visible spectrometer. Fourier transform infrared (FTIR) analysis of ZnO/SnO$_2$ was recorded with a Thermoscientific Nicole 6700 analyzer in 4000–400 cm$^{-1}$ range.

**Result and Discussion**

**X-ray diffraction analysis.**—XRD analysis of ZnO/SnO$_2$ nanosstructure is shown in Fig. 3a. All the peaks of the sample exactly match with JCPDS No. 79–0207 for ZnO hexagonal form and JCPDS No. 21–1250 for SnO$_2$ tetragonal structure. The average

![Figure 1. Schematic Diagram of TENG.](image)

![Figure 2. Flow chart for the synthesis of ZnO/SnO$_2$ heterostructure.](image)
The crystallite size of all the peaks was calculated using Eq. 1

\[ D_{\text{hkl}} = \frac{0.9\lambda}{\beta \cos \theta} \]  

[1]

Here \( \lambda \) is the incident X-ray wavelength, \( \theta_{\text{hkl}} \) is known as the Bragg diffraction angle and \( \beta_{\text{hkl}} \) is known as the full width at half maximum (FWHM) of all the peaks in XRD pattern. The value 0.9 is the shaping factor and depends on the shape of the crystalline material. The average crystallite size of the material was found to be 17.59 nm. The interplanar distance values were observed less in comparison to standard data.

The average size and strain found in grains were analyzed by Williamson and Hall (W-H) plot. Figure 3b shows the W-H plot with a slope of \( 2.37 \times 10^{-3} \), which denotes the compressive microstrain in ZnO/SnO\(_2\). From the intercept value of W-H plot the average crystallite size was found to be 16.58 nm, which is nearly equivalent to the value from the Scherrer formula (17.59 nm).

**Nanozetasizer analysis.**—Nanozetasizer was used to calculate the particle size of synthesized ZnO/SnO\(_2\) nanomaterials, which works on the principle of dynamic light scattering (DLS). In this method, particles were assumed to be spherical.

The real-time graph was obtained from the instrument to tabulate particle size shown in Fig. 4a. It can be visualized from Fig. 4a that the diameter of the material lies between 50–80 nm and the average size of the particle was 63.23 nm.

Zeta potential is defined as the voltage at the edge of the shipping plane with respect to the dispersion medium. With the help of zeta...
Figure 6. FTIR spectrum of ZnO/SnO₂ nano heterostructured material.

Figure 7. SEM micrographs of ZnO/SnO₂ heterostructured film (a) at 1.0 μm scale (b) at 0.5 μm scale, (c) EDS spectrum along with (d) elemental composition, and (e)–(h) elemental mapping of all elements present in ZnO/SnO₂.
potential, we can predict the long-term stability of dispersed material. From Fig. 4b, it can be visualized that the zeta potential and conductivity of the sample were $-10.3 \text{ mV}$ and $0.0116 \text{ mS cm}^{-1}$ respectively in isopropyl alcohol at 25 °C.

**UV-visible spectroscopy analysis.**—The absorbance of the UV-Vis spectrum in the range (250–800 nm) for synthesized ZnO/SnO$_2$ is shown in Fig. 5 with excitation wavelength at $\sim 363 \text{ nm}$. The relation given in Eq. 2 is known as Tauc’s relation, which is used to obtain band gap.

$$\alpha = \frac{A(h\nu - E_g)^n}{h\nu}$$

Here $h\nu = \text{photon energy}$, $\alpha = \text{absorbance coefficient}$, $A = \text{characteristic coefficient}$, $h = \text{Planck’s constant}$, and $n = \text{power factor}$. Power “$n$” is dependent on the type of transition, and “$n$” is $\frac{1}{2}$ because of the direct allowed transition. The calculated value of band gap is $\sim 3.49 \text{ eV}$.

It is well established fact that when the size of bulk material is reduced, its band gap increases. The higher the band gap, smaller the size and hence the larger is surface to volume ratio. Higher the surface-to-volume ratio, the higher will be the interaction with analyte. This will enhance the sensing performance.

**FT-IR spectroscopic analysis.**—FT-IR spectroscopy is an operative tool for the investigation of functional groups existing in nanomaterials. The peaks in the region between 400–700 cm$^{-1}$ are associated with M-O (metal-oxygen) and M-OH (metal-hydroxide) bonds. The FTIR spectrum of ZnO/SnO$_2$ heterostructure is visualized in Fig. 6. The peaks of Zn-O and Sn-O confirmed the development of ZnO/SnO$_2$ nanocomposite at 636 and 478 cm$^{-1}$ respectively. The small peaks at 1644 cm$^{-1}$ contributed to C=O vibration through stretching. The peaks at 3440–3450 cm$^{-1}$ range are accredited to O–H stretching vibrations, specifying the existence of water molecules in the samples.

**Surface morphological study.**—The surface morphological study of the ZnO/SnO$_2$ nanostructure thin film was carried out at different resolutions using SEM as shown in Figs. 10a and 10b.

It can be visualized from these figures that the spherical-shaped particles formed a cluster in which voids can be visualized. In Fig. 10c elemental analysis by energy dispersive X-ray spectroscopy is shown. It can be seen that Sn, O, and Zn are present in synthesized material. In Fig. 7d % weight and atomic weight are shown. Elemental mapping was carried out to demonstrate the distribution of elements on the surface of synthesized heterostructured ZnO/SnO$_2$. It can be evidently visualized from Figs. 7e–7h that Zn and Sn were successfully merged together to form heterostructure along with oxygen.

**Fabrication of PET-based TENG.**—TENG structure is fabricated by PET sheet, copper tape with 2 mm thickness, Kapton tape, bi-sided spacer foam tape, and ZnO/SnO$_2$ nanoparticles$^{29,30}$ prepared by hydrothermal method. A flow chart of fabrication is given in Fig. 8. It is worth mentioning here that no sophisticated methods for coating such as CVD, PVD, etc. were used in the device manufacturing so that the device can be used in plug-and-play mode (within 25 min). 2 copper-foil tapes which act as an electrode were joined to PET sheets. One of them was attached to Kapton tape. To improve the triboelectric effect, Kapton tape was rubbed with rough PET, Copper, Kapton, Epoxy ZnO-SnO$_2$ composite layers.
and fine sand paper. Another PET sheet was drop cast with epoxy ZnO/SnO$_2$ blend. The heterostructure of ZnO/SnO$_2$ composite enhances the inner electric field within the nanoparticle. The used epoxy was composed of two components: (1) Resin and (2) Hardener. Both components were mixed in a 3:2 ratio and ZnO/SnO$_2$ nanoparticles were poured into this epoxy. This mixture was properly mixed with a spatula and then one PET sheet was drop cast with this epoxy mixture. Later, this coated PET sheet was dried at room temperature (38 °C) for 12 h. The adhesive strength of the epoxy nanocomposite film was found to be very good. A schematic diagram of assembled TENG can be visualized in Fig. 9. The effective contact area of 30 cm$^2$ of TENG was separated by spacers as shown in Fig. 10.

**TENG testing and self-powered LPG sensing.**—The following components were used to test the performance of fabricated TENG:

a) Keithley 6517B electrometer with GPIB interface
b) Owon SDS-1102 Digital Oscilloscope

Variation of voltage with time in respect of mechanical triggering, voltage generates up to 75 volts which are good for

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**Figure 10.** Image of fabricated TENG.

**Figure 11.** Plotted graph of output (a) AC voltage and (b) AC current using Keithley electrometer.

**Figure 12.** Schematic diagram of self-powered sensing setup.
operating many electronic devices. For a sensor device, the output of the device (TENG) works as voltage source $V_{IN}$. $R_1$ acts as the internal resistance of the circuit, while $R_2$ represents the sensing film. The voltage drop across $R_2$ is:

$$ V_{OUT} = \frac{R_2}{(R_1 + R_2)} \times V_{IN} \quad [3] $$

Equation 3 clearly shows that output voltage across $R_2$ is directly proportional to the value of resistance $R_2$, i.e., with the change in the magnitude of the $R_2$, the magnitude of the $V_{OUT}$ is also changed. The mechanism of self-powered sensors can be easily understood by the above equation.

Figure 11 presents the output of fabricated TENG (a) AC voltage and (b) AC current using Keithley electrometer. Here output AC and DC voltages are ~75 volts which is higher than the paper-based TENG. Thus this PET-based and made of ZnO/SnO$_2$ nanocomposite is better than the previously developed TENG.

The mechanism may be understood as when two dielectric materials Kapton and epoxy ZnO/SnO$_2$ nanocomposite contact each other, then the phenomenon of electrification generates static charges that appear on the outer surface of both triboelectric layers. When we tap the TENG, cation generates static charges that appear on the outer surface of the dielectrics. These cations then create an electrostatic field between the two dielectrics. This electrostatic field then attracts the free electrons from the metal layer to the material surface in the form of an AC voltage.

The self-powered sensing setup shown in Fig. 12 was assembled as described in previously reported articles.33,34 By Eq. 3, it is confirmed that when the resistance of the sensing film reduces, the voltage drop across this film also reduces by Ohm’s law and vice versa. For this, ZnO-SnO$_2$ resistance-based sensing was performed under 0.5, 1.0, and 1.5 vol% concentration as shown in Fig. 13. When LPG was put in the air, oxygen gas molecules gets adsorbed on the film to form O$_2^-$ ions, which generates the base resistance in the air.35,36 When LPG is put onto the material, it primarily gets contact with the O$_2^-$ layer. H$^+$ ions from LPG react with O$_2^-$ to produce the following species as shown in Eq. 4:37

$$ C_nH_{2n+2}(LPG) + O_2^- \leftrightarrow C_nH_{2n}O + H_2O + e^- \quad [4] $$

This electron from the above reaction reacts with holes in the material, thus total charge carrier concentration diminishes. This causes an increment in the resistance as shown in Fig. 13. When LPG was expatriated from the chamber, the total number of H$^+$ molecules declined and more and more environmental oxygen comes in contact with the sensing film and produces an O$_2^-$ coating again.7,8,28,32–34,36,37 Thus, the resistance again reaches its initial condition. Now, this sensing film was powered by TENG as discussed above and voltage drop across the film was measured as shown in Fig. 14. The voltage drop increases as the concentration of LPG increases as shown in Fig. 14. A comparative table of previously reported LPG sensor is shown below:

The maximum output voltage in air, 0.5, 1.0, and 1.5 vol.% were 3 volts, 12 volts, 33 volts, and 72 volts respectively. The sensor response was calculated by the ratio of voltage at a particular gas concentration and voltage in the air.33 The observed sensor response for air, 0.5, 1.0, 1.5 vol.% were 1, 4, 11, and 24 respectively. Nanogenerator-based self-powered sensors are better than conventional resistive sensors because there is no use of batteries in self-powered sensors. The lifetime of self-powered sensors is far better than resistive sensors. Further, the response and recovery times of the self-powered sensor are much smaller (Quick responsive), because of the use of rapidly changing pulse type input voltage. Table I shows the comparative analysis of resistive sensors with the self-powered sensor.

The present material is highly selective for LPG as compared with other gases like acetone, ethanol, and ammonia as shown in Fig. 15. There are two mechanisms that can be regarded on the involvement of high selectivity of the ZnO-SnO$_2$: (1) Effect of depletion layer on the target gas, and (2) Effect of LUMO (Lowest Unoccupied Molecular Orbital) energy.42 It is evident from the LPG sensing response, the surface of the sensing component is enhanced with reactive surface electrons (e$^-$) which readily react with oxygen from the environment. The oxygen then gets ionized and adsorbed on the material surface in the form of O$_2^-$ ions. It makes the surface lack of free electrons and the adsorbed O$_2^-$ ions act as a sensing layer of the material for target gas. When a reducing gas reacts with this layer, the gas molecules contribute electrons to the material by reacting with the adsorbed O$_2^-$ ions. If the gas is oxidizing in nature, it has a tendency to take electrons from the surface of p-type materials. But the surface is now electron lacking. To deliver the energy mandatory for the complete desorption of O$_2^-$ ions, the material needs to be heated. Hereafter in case of our material, it is very tough for the oxidizing gases (acetone, CO$_2$) to respond at room temperature.

Another well tested aspect for selectivity is the LUMO energy. The tested orbital energies of ethanol, methanol, acetone, ammonia, propane and butane were documented as, 0.12572 eV, 0.19728 eV, 0.20525 eV, 0.11887 eV, 0.07647 eV and 0.06717 Hartree respectively.82–84 The molecules with lower value of LUMO energy are effortlessly sensed at room temperature, while the molecules having higher value of LUMO energy require higher operating temperature (~280 °C–362 °C) for a good response. This is based on the fact that the abilities of charge carrier transmission is inversely proportional to the LUMO energies.42

The maximum sensor response for acetone, ethanol, and ammonia are 1.13, 1.03, and 1.5 respectively.

The minimum recovery and response times for 0.5 vol% was observed as shown in Figs. 14e and 14f. The response and recovery times were found to be 120 ms and 135 ms respectively. Such a low response and recovery time shows that the fabricated device is ultra-fast.
Conclusions

Miniaturization is the beauty of nanogenerators. The generation of power needs to be updated with inventions. The fabricated TENG generates a maximum output voltage of $\sim 75$ volts which is more than the output of paper-based TENG. This TENG was used as a power source to operate a resistive LPG sensing film. The maximum response of 24 and minimum response-recovery times of 120–135 ms were observed, which makes this LPG sensing device ultra-fast. Further research in this field can save the non-renewable source of energy. The invention of nanogenerators may prove to be the beginning of a new stage in the history of mankind. These can be used for harvesting mechanical energy; self-powered nanosystems, solar energy, nano-piezotronics, Bio-interfacing, bio-inspired fabrication & bioengineering. However, further enhancement in electrodes by CVD and PVD methods and by using highly conducting structures like CNT, ITO-coated PET may further enhance the output electrical performance of TENG. This can be used for sustainably powering biomedical microsystems as well as fully integrated transducers that are needed for autonomous sensor networks.

Prabhakar Yadav: Writing—original draft, synthesis, Characterization, Investigation, Methodology. Ajeet Singh: Writing—review & editing.
| S. No. | Detecting material                                      | Target gas (Room Temperature) | Sensor response \((R_g/R_a)\) | Sensor response% \((\lvert R_g-R_a\rvert/R_a*100)\) | Res. time | Rec. time | References |
|-------|--------------------------------------------------------|------------------------------|-------------------------------|-------------------------------------------------|-----------|-----------|------------|
| 1     | ZnSnO<sub>3</sub>/ZnO                                 | LPG                          | —                             | 83.23                                           | 88 s      | 72 s      | 38         |
| 2     | Pd decorated ZnO                                      | H<sub>2</sub>                 | —                             | 373                                             | 100 s     | —         | 39         |
| 3     | SnO<sub>2</sub>/ZnO                                    | H<sub>2</sub>                 | —                             | 471                                             | 350 s     | 150 s     | 40         |
| 4     | Pd-coated ITO/PET                                     | H<sub>2</sub>                 | 1.5                           | —                                               | 30 min    | 15 min    | 41         |
| 5     | Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> nanohybrid | LPG                          | 1.98                          | —                                               | 34.39 s   | 45.17 s   | 35         |
| 6     | Paper/ZnO-SnO<sub>2</sub> heterostructure              | LPG                          | 24                            | 2300                                            | 120 ms    | 135 ms    | Present paper |

Table I. Comparative table of previously reported LPG sensors.
Synthesis, Characterization, Investigation. **Shakti Singh**: Validation, Visualization, Data curation, Investigation, Supervision. **Dheeraj Kumar** : Data curation, Validation.

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**Statements & Declarations**

The authors declare that no funds, grants, or other support were received during the preparation of this manuscript.

**Competing Interests**

The authors have no relevant financial or non-financial interests to disclose.

**Data Availability**

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

**ORCID**

Shakti Singh [https://orcid.org/0000-0001-7977-7178](https://orcid.org/0000-0001-7977-7178)

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