Synthesis and Characterization of Self-healing Nano-Materials and their Applications

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Summary

Since the last few decades, the branch of nanotechnology has been thriving with a huge intensity [1]. The nanomaterials display diverse physical and chemical habits than the bulk material because of their shape and size. Other than the size effect, "Quantum confinement" also shows a crucial role in showing amazing properties in nano regime [2].

Self-healing nanomaterials are currently one of the trending research areas in nanoscience and nanotechnology. Self-recovering or Self-healing nanomaterials are the materials, which can redevelop their structure after deformation by means of environmental or mechanical causes [3]. Self-healing property is nothing but the mimic of the healing functionality found in some creatures like star fish and lizard tail [4,5]. Self-healing constituents are used nowadays in various kinds of electronic devices like solar cell, sensors, batteries, etc [6]. As a result of frequent environmental weathering and mechanical pressure, internal stress arises, which leads to degradation of appropriate functioning of these electronic

materials by the time [7]. In electronic devices, by using the self-healing nanomaterials the output characteristics of the device can be alleviated throughout a long period i.e. after deformation these electronic devices can re-claim their original functionality by self-generation behaviour. These materials are of two types: **(1)** Intrinsic self-healing materials, and **(2)** Extrinsic self-healing materials [7–9].

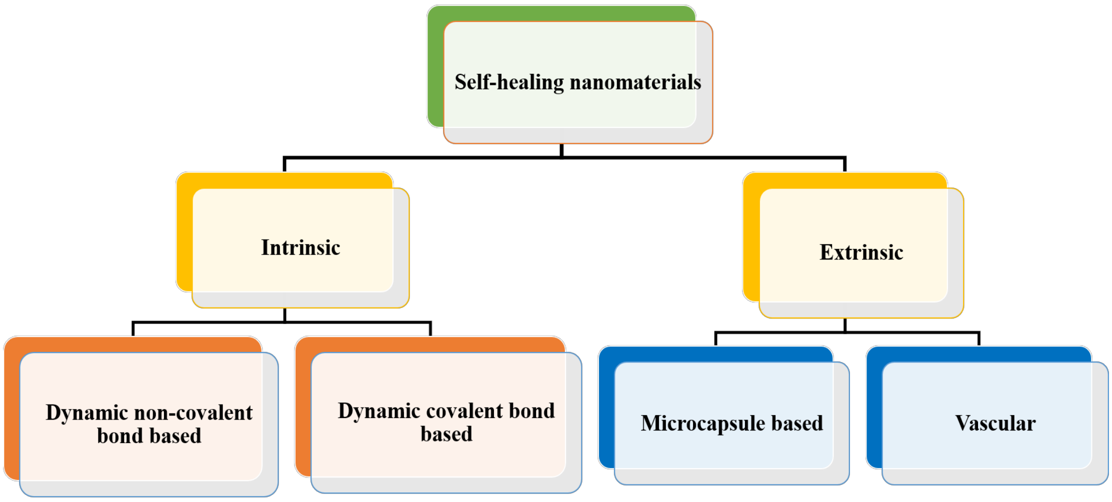

Fig. 1 Types of self-healing nanomaterials

Intrinsic SHNs contain of reversible non-covalent and covalent bonds, regeneration of which gives renovation of the polymeric assembly afterwards deformation [9]. These self-healing materials are also of following two types:

(a) Based on Dynamic non-covalent bond: In this type of intrinsic healing, renovation is administered by metal coordination bonding, hydrogen bonding, electrostatic cross-linking, etc [7].

(b) Based on Dynamic covalent bond: In this type of intrinsic self-healing, healing is associated to the bonds which are able to be originated among dissimilar atoms and are kinetic in the influence of an external entities. Many kinds of dynamic covalent bonds like acyl hydrazone, Diels–Alder reaction, ester, disulphide, olefin, and imine are explored for the synthesis of intrinsic self-healing nanomaterials. As compared with the noncovalent bonding, external forces like heat, pH, pressure, and light is always required to activate the healing procedure.

In extrinsic self-healing, healing procedure necessitates the exhaustion of healing agent which are pre-placed during the production [9]. This self-healing practice is labelled as "extrinsic self-healing" because healing phenomenon is not intrinsic property of these material, but some external agent is required to rapture the healing container inside matrix. This is of following two types:

(a) Based on Microcapsules: In this type of extrinsic self-healing, micro capsule with micro or nano healing agents are incorporated in the interior of the polymer. Within these microcapsules regenerating agent are filled, that is conveyed towards the impaired region prior to fracture of the capsule.

(b) Based on fibres: In this type of extrinsic self-healing, the healing material is filled inside a vein like structure inside a system.

Mainly oxidative polymerization, micro emulsion, sol-gel and direct mixing methods were employed to synthesize various types of self-healing materials.

These self-healable nanomaterials were further utilized to fabricate self-healable and self-powered sensing devices. The sensor is an instrument that perceives the changes in a physical variable and generates an output as a function of that particular input [10–12]. These self-powered sensing devices are based on Triboelectric nanogenerator. These devices (Piezoelectric and triboelectric nanogenerators) are able to produce electrical energy from mechanical energy by different means [13–20]. Because of the repeated mechanical force, the internal deformation occurs inside these self-powered sensing devices. This is the reason self-recovering materials are used during fabrication of these sensors for stable output for a long duration [21,22]. All the Chapters are summarised as follows:

1. **Chapter 1** deals with the description of the self-healing materials, their types, self-healing process, properties and synthesis method. Various types of self-healing materials along with their healing mechanism was discussed in this chapter. A comprehensive literature investigation of previously reported self-healable materials with their mechanism was also presented in this chapter. Further various synthesis techniques were also described, which are used to synthesize such materials. A detailed literature investigation of earlier reported

research in the field of self-healable electronic devices are discussed in this chapter. This chapter also describes different application of self-healing nanomaterials in electronics devices. This chapter describes various types of self-powered devices based on nanogenerators in which self-recovering materials are used. The difference between self-powered sensors with resistive sensors is also demonstrated in this chapter. Discussion of the working mechanism of different sensors like photo, gas and fluoride sensors, which are performed to demonstrate self-healable sensing devices are discussed. This chapter illuminates the main problems emerged in the earlier reported studies.

2. **Chapter 2** deals with a procedure to prepare a dual layered photo detection sensor, which can reoccupy its actual sensing performance after damage occurs in its physical structure. In this work two layers were used, microcapsule based bottom layer and upper NZF sensing layer. The NZF nanomaterial was synthesized via citrate acid aided sol-gel method, and was used as the resistive sensing film. Microcapsules of U-F infused with flaxseed oil and NZF core were prepared by oil emulsion method, and used as a self-healing material. Via free-flowing skill of flaxseed oil, NZF nanoparticles are able to fill in the damaged areas when the microcapsules are ruptured. In this layer flaxseed oil acts as a transporting agent, which carries NZF nanoparticles towards the cracks. The preparation methods are very easy, simple and inexpensive. Different characterization techniques were employed to realize the thermal stability, chemical bonding, elemental analysis, optical band gap, crystal information of the synthesized materials. The polycrystallinity along with purity of NZF were established by XRD investigation. The encapsulation of NZF inside U-F polymer was proved by TEM and SAED and FTIR analysis. Via TEM analysis, the average NZF dimension can be found as ~20 nm.

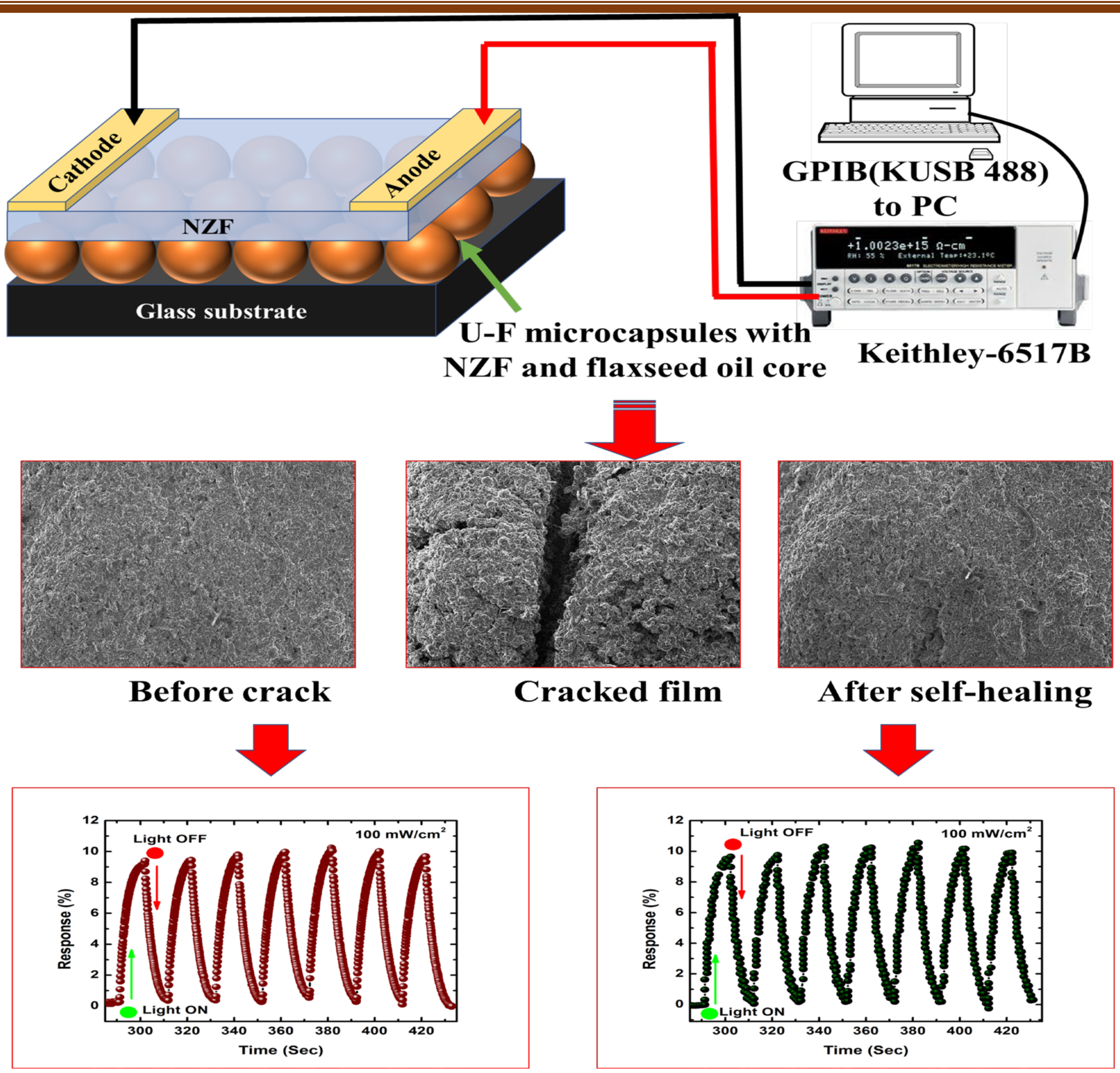

Fig. 2 The schematic presentation of Bilayered ultra-responsive self-healable photodetector along with set-up and healing performance

The measured band gap of NZF via Tauc plot in UV-Visible analysis was ~3.51 eV. The effective restoration of film was confirmed by SEM and EDS analysis. By regaining the original structure, the sensing element was competent to reinstate the sensing properties. The response/recovery time of the uncracked sample were 1.74/3.28 sec, while those for the healed sample were 1.75/2.84 sec. The most important parameter of healing efficiency was calculated, which tells the restoration of electrical properties of fabricated photo detector. The sensing capability of the healed sample was reproducible up to 98.5%. These outcomes approve that a photodetector is successfully prepared that can re-establish its sensing properties after any mechanical damage.

3. **Chapter 3** describes the demonstration of self-healable and self-powered visible light photo detector. In this Polyaniline-Polypyrrole (PANI-PPY) was produced using low temperature aided oxidative polymerization method. The method of preparation is very simple to form nano-dimension (~30 nm) 2-D NFs. A high-resolution SEM image of NFs confirms the smallest width of NF ~30.35 nm. The measured band gap of PANI-PPY via Tauc plot in UV-Visible analysis was ~1.85 eV. The NFs formation was also explained in this chapter via thermal scission and surfactant effect. The goal of this work is synthesis of self-healing nanomaterial and its application in self-powered photo sensor.

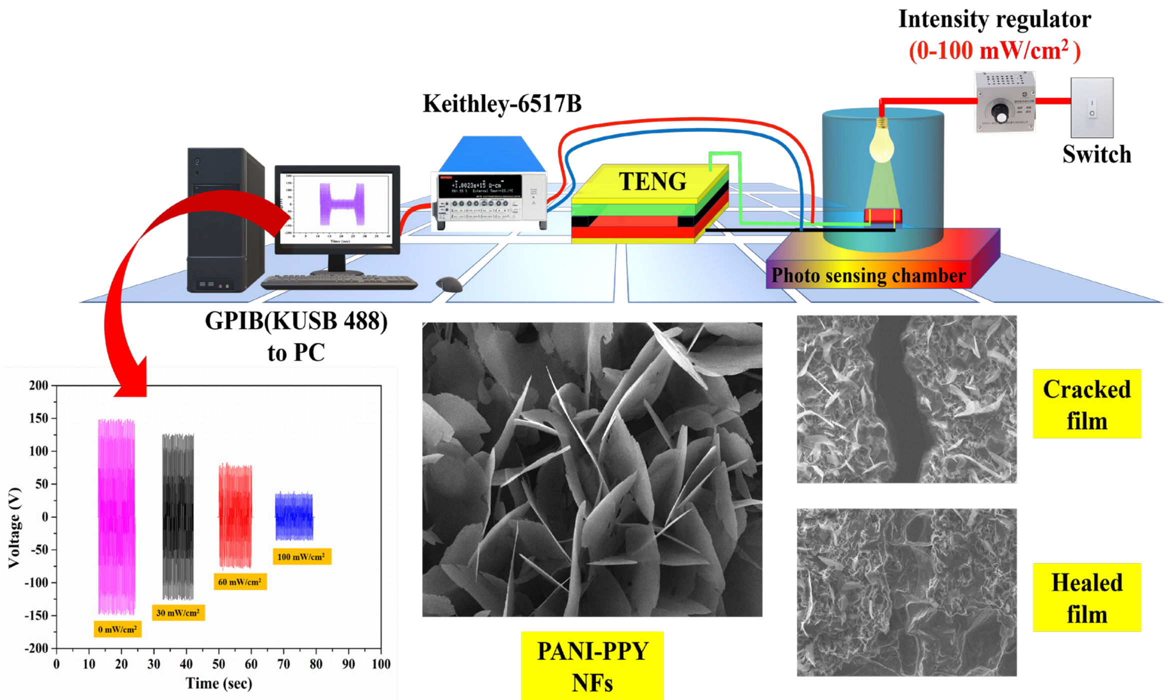

Fig. 3 The schematic presentation of 2-D PANI-PPY based self-healable and self-powered visible light photo detector

Self-healing ability was absent in earlier published self-powered photo sensors that can change the output enactment of the device after damage. This work emphases on advances of self-recoverable and self-powered photo sensor. Self-healing ability was absent in earlier published self-powered photo sensors that can change the output enactment of the device after damage. TENG measurements was taken to explore the performance of TENG. A maximum voltage of 149 volts, maximum current of 16 μA, maximum current density of 0.56 μAcm^{-2}, and maximum power density 83.56 μWcm^{-2} were detected from the sensing device that illustrates greater performance against the previously testified self-powered sensors. The least response/recovery times were observed for 30 mW/cm^2, which are 0.41 sec and 0.45 sec. The % response at 0, 30, 60 and 100 mW/cm2 illumination were observed to be 0, 17.78, 78 and 278 % respectively with sensitivity 2.78 %response/mWcm^{-2}. Most significantly the TENG doesn't display any foremost variations in voltages pre and post healing. The most novel characteristic of this research is acetone supported rapid self-restoration presented in Polyaniline-Polypyrrole (PANI-PPY) nanoflakes. The maximum healing efficiency of 99.8% was accomplished in this work.

4. **Chapter 4** deals with the a self-powered, self-healable, and visible light-enhanced LPG sensor based on PANI-PPY (2P) 2-dimensional nano-sheets.

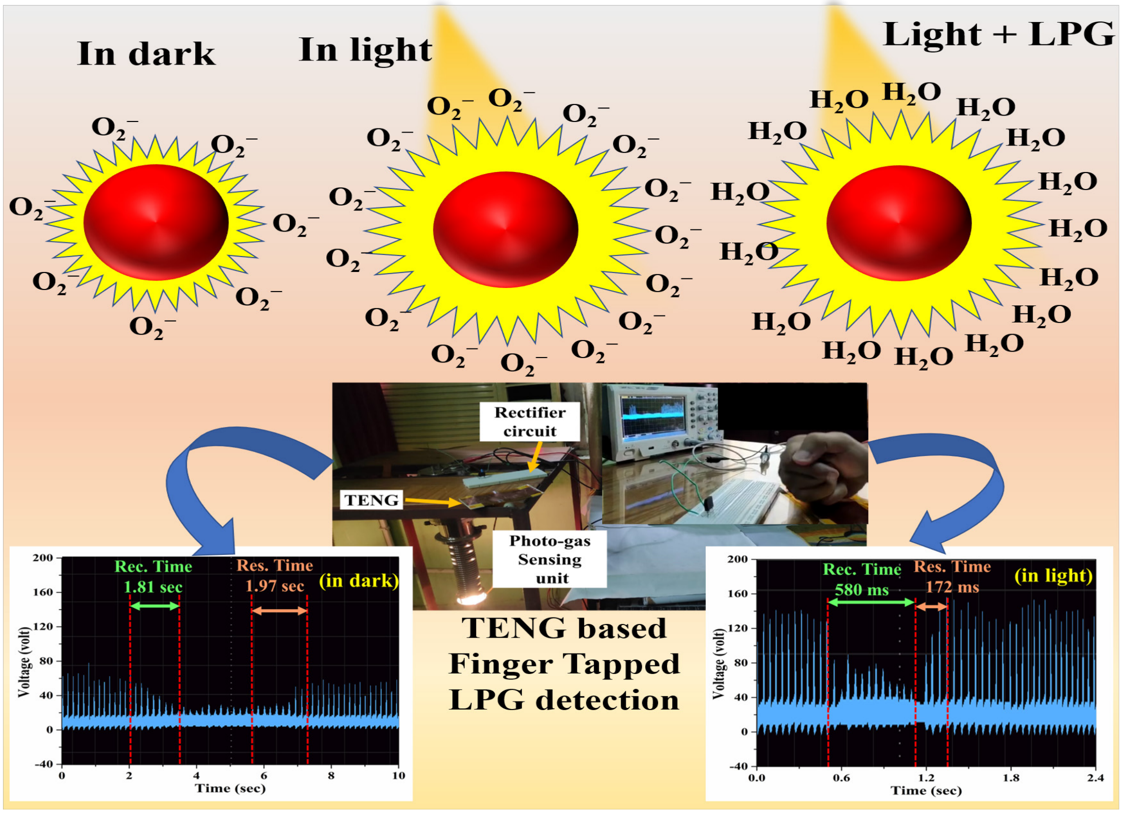

Fig. 4 The schematic presentation of 2-D PANI-PPY based self-healable and self-powered visible light stimulated LPG sensor

The procedure to synthesize PANI-PPY (1:4) was similar to what followed in chapter 3 except the ratio of monomer. The copolymer was thoroughly investigated using different characterization techniques. Multiple nano-flakes can be found all through the sample with minimum thickness of ~24.76 nm. HRTEM analysis was performed to authorize 2-dimensional polymer structure. FTIR, RAMAN, XRD and TGA analysis were also performed to characterize the sample. The measured band gap of PANI-PPY via Tauc plot in UV-Visible analysis was ~2.02 eV. The BET analysis confirms the specific surface area, avg. pore size, and mean pore volume of ~125.6 m^2/g, 30.23 nm, and 0.086 cm^3/g respectively. The material showed brilliant LPG detection below its LEL, when illuminated with visible light at room temperature. The sensor was operated with TENG (3P) that generated an output voltage and current of ~142 volt and 80 μA respectively. Using the output of TENG as power source, the sensing element showed a maximum sensor response ~94.67 and sensitivity ~52.67 SR/vol.% under the influence of 30 mW/cm^2 visible light. The material was able to detect very low concentrations of LPG almost instantaneously with response/recovery times 172/580 ms. Self-healing ability was also successfully introduced in the TENG layer to regain its properties after any damage. The Maximum healing efficiency of ~99.7% was achieved here in this work.

5. **Chapter 5** deals with the a unique, cost-effective, and quick detection method of fluoride ions in water. The device is fabricated using waste materials like BSR rubber, used polythene bag, and used plastic sheet, which validates the waste to energy conversion ability with maximum output voltage of ~242 volts and 40 μA current. A new technique is used and validated to detect fluoride ions, using a TENG based device fabricated using hazardous waste (BSR). BSR decorated with Lanthanum doped Polyaniline-Polypyrrole (LaPP) nanospheres (BSR-La) mixed in PDMS (PDMS/BSR-La) was used to form one layer of the TENG that is self-healable in nature. LaPP nanomaterial with perfect spherical shape within a size 14-50 nm was confirmed by SEM analysis. The elements present in the LaPP were confirmed by both EDX and elemental mapping. FTIR, XRD and UV-Visible analysis were also performed to characterize the sample.

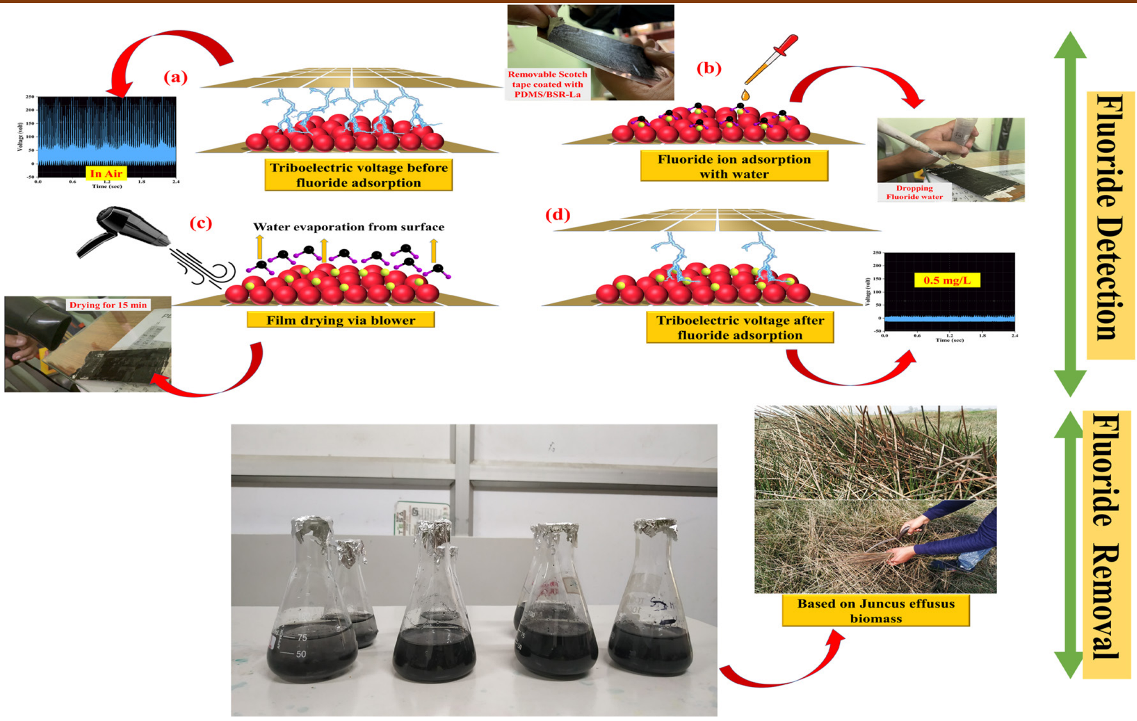

Fig. 5 The schematic presentation of 2-D PANI-PPY based self-healable and self-powered visible light stimulated LPG sensor

The optical band gap measured from the Tauc plot was ~1.58 eV. The present chemisorbed-based self-powered fluoride detection device shows a maximum sensor response of 12.10, % sensor response of 1110 %, and limit of detection of 4.7 µM, which is very high. The self-healing ability of PDMS/BSR-La nanocomposite shows fast healing in ~25 min with a healing efficiency of 99.9%, which is one of the unique features of this device. Besides that, this manuscript also demonstrates the method of resistance measurement to minimize the water effect and corresponding output voltage behavior of the sensor. In summary, this work establishes the exclusive way to detect fluoride using waste materials.

6. **Chapter 6** gives the concluding remark and comparative analysis of all synthesized materials and fabricated devices. This chapter deals with the summarised information of the material characteristics, sensing output, and future scope of all the chapters mentioned previously. A chapter wise overview of this thesis including the materials, their application, sensor response, presence of self-healing and self-powered ability along with response/recovery times is depicted in the Table 1 as follows:

Table 1 Chapter wise information and characteristics of developed material and sensor.

Chapter No.	Sensing element	Application	Max. Sensor response	Self-healing ability and efficiency	Self-powered ability	Min. Response time	Min. Recovery time
2.	**$Ni_{0.2}Zn_{0.8}Fe_2O_4$@ poly (Urea-Formaldehyde) microcapsules**	Photo sensor (Visible light)	11	Present (98.5%)	Not present	1.74 sec	3.28 sec
3.	**2-D Polyaniline-Polypyrrole Nanoflakes**	Photo sensor (Visible light)	3.78	Present (99.8 %)	Present	0.41 sec	0.45 sec
4.	**2-D Polyaniline-Polypyrrole Nanosheets**	Visible light induced LPG sensor	94.67	Present (99.7 %)	Present	172 ms	580 ms
5.	**Lanthanum doped Polyaniline-Polypyrrole nanospheres**	Fluoride ion sensor	12.10	Present (99.9 %)	Present	–	–

In conclusion, both physical and chemical sensor with self-restoring and self-powering capability were successfully demonstrated. Besides this, A unique new method to detect fluoride ion well below permissible limit was demonstrated with self-healing and battery less ability.

Future scope of research work

- Other functionalities like self-cleaning, shape memory etc. along with healing and self-powering may be incorporated in a single device.
- Other polymers with different nano dimensions like 2-D, 0-D, 1-D may be in explored and incorporated in TENG for better performance.
- Other polymers and materials may be explored for enhanced self-healing ability.
- Other types of chemical, gas and biological sensor like CO_2, ammonia, different acids may be explored with PANI-PPY nanostructures.
- Further studies of nanostructured polymer and formation mechanism can be explored.
- Effects of different synthesis parameters like precursors, concentrations, solvents, pH, temperature etc. on the prepared material can be studied further for optimization.

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List of Publications

Part of the thesis published and communicated in the refereed journals:

1. **S. Singh,** A. Bhaduri, R.K. Tripathi, K.B. Thapa, R. Kumar, B.C. Yadav, Improved sensing behaviour of self-healable solar light photodetector based on core-shell type $Ni_{0.2}Zn_{0.8}Fe_2O_4$@ poly (Urea-Formaldehyde), Sol. Energy. 188 (2019) 278–290.
2. **S. Singh,** R.K. Tripathi, M.K. Gupta, G.I. Dzhardimalieva, I.E. Uflyand, B. Yadav, 2-D self-healable polyaniline-polypyrrole nanoflakes based triboelectric nanogenerator for self-powered solar light photo detector with DFT study, J. Colloid Interface Sci. 600 (2021) 572–585.
3. **S. Singh**, P. Yadav, M.K. Gupta, G.I. Dzhardimalieva, J. Yoon, C. Maiti, B.C. Yadav, Gigantic stimulation in response by solar irradiation in self-healable and self-powered LPG sensor based on triboelectric nanogenerator: Experimental and DFT computational study, Sensors Actuators B Chem. 359 (2022) 131573.
4. **S. Singh**, C. Bhan, M.K. Gupta, J. Yoon, C. Maiti, J. Singh, B.C. Yadav, Waste Material Based Self-Healable and Self-Powered Detection of Hazardous Fluoride Ions and its Removal with Novel Adsorbent: An Unexplored and Highly Responsive Detection Method, Journal of Hazardous materials. **(Under Review)**

Work not included in the Thesis

1. A. Bhaduri, **S. Singh**, K.B. Thapa, B.C. Yadav, Improved room temperature liquefied petroleum gas sensing performance of $Ni_{0.5}Zn_{0.5}Fe_2O_4$@Cl-doped polypyrrole nanoweb, Mater. Sci. Eng. B. 279 (2022) 115660.
2. A. Bhaduri, **S. Singh**, K.B. Thapa, B.C. Yadav, Visible light-induced, highly responsive, below lower explosive limit (LEL) LPG sensor based on hydrothermally synthesized barium hexaferrite nanorods, Sensors Actuators B Chem. 348 (2021).
3. G.I. Dzhardimalieva, B.C. Yadav, **S. Singh**, I.E. Uflyand, Self-healing and shape memory metallopolymers: State-of-the-art and future perspectives, Dalt. Trans. 49 (2020) 3042–3087.
4. A. Bhaduri, **S. Singh**, R.K. Tripathi, U. Kumar, K.B. Thapa, B.C. Yadav, Healable, highly sensitive LPG sensor based on $Ni_{0.4}Zn_{0.6}Fe_2O_4$ nanohybrid grown by auto combustion process, Sensors Actuators B Chem. 327 (2021) 128840.

5. S. Sikarwar, **S. Singh**, Satyendra, R. Srivastava, B.C. Yadav, V. V. Tyagi, Design and development of lab model of piezo-optic sensor for Structural Health Monitoring, Smart Mater. Struct. 26 (2017) 105047.
6. S. Sikarwar, Satyendra, **S. Singh**, B.C. Yadav, Review on pressure sensors for structural health monitoring, Photonic Sensors. 7 (2017) 294–304.
7. A. Singh, **S. Singh**, B. C. Yadav, Gigantic enhancement in response of heterostructured CeO_2/CdS nanospheres based self-powered CO_2 gas sensor: A comparative study, Sensors Actuators B Chem. **(Under review)**
8. P. Yadav, A. Singh, **S. Singh**, D. Shukla, Design and development of Paper/ZnO-SnO_2 heterostructured ultra-fast TENG based LPG sensor, ECS Sensor plus. **(Under review)**

Papers presented in National and International Conference/Webinar/Workshop

1. National Conference on "Sukshm Padarth evam Sambaddh Chetan Urja" 1^{st} – 3^{rd} February, 2019, BBAU, Lucknow. **(Oral Presentation)**
2. International Webinar on Nanoscience and Nanotechnology (IWNN-2020), 27^{th} – 29^{th} November, 2020, BBAU, Lucknow **(Oral presentation)**.
3. International Conference on Frontiers in Physics, Materials Science & Nanotechnology (FPMSN-2022) March 25-26, 2022, Sirsa **(Oral presentation).**
4. International Conference on Recent advances in Science (ICRAS-2022), 1^{st}-2^{nd} April, 2022, Bareilly **(Oral Presentation)**.
5. WEBINAR on 'SCIENCE: In Today's Prospect' February 28, 2022, Siddharthnagar **(Oral Presentation)**.
6. International Conference on Nanoscience and Nanotechnology (ICNN-2017), 22^{th} – 24^{th} September, 2017, BBAU, Lucknow **(Poster presentation)**.
7. "Synergistic Training Utilising the Scientific and Technological Infrastructure" (STUTI) training workshop, 22-28 August, 2022, BBAU, Lucknow **(Workshop)**

Achievements

- Project fellow in Indo-Russian project-2019 (Reference no. INT/RUS/RFBR/P-375).
- 13 days working in IPCP- Russian Academy of Sciences, Chernogolovka, Russia for synthesizing self-healable polymers and Advance polymerization synthesis technique under Indo-Russian project-2021
- 10 days of research work in CSIR-AMPRI (Bhopal) for developing hybrid nanogenerators for self-powered sensing applications.
- Best oral presentation at International conference on Recent Advances in Science (ICRAS-2022), Bareilly.
- Best oral presentation at webinar on National Science Day-2022, Lucknow.
- Best oral presentation at International webinar on Nanoscience and Nanotechnology (IWNN-2020), Lucknow.
- Best poster presentation at International conference on Renewable Energy for Sustainable Environment: Challenges and Remedies (ICRESE-2017), Jammu.