Detection of high sensitive acoustic region for sensible applications of electrospinning based PVDF-TrFE nanofiber sensor

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Abstract
Cardinal amount of assessment has been vested on using Polyvinylidene Fluoride-co-Trifluoroethylene (PVDF-TrFE) electrospinning based nanofibers for subtle applications as acoustic sensor. In the current course of experiment, utilizing electron, proton and ionic conductive properties of the PVDF-TrFE co-polymers in conjunction with aluminum (Al) and copper (Cu) as electrodes, demonstrates strong acoustic to electric conversion ability within the audible frequency range of 30 Hz to 15 KHz and fortuitously, sensitive acoustic region has been commended for intricate medical applications. Furthermore, enhanced sensitivity was observed due to advance charge polarization by using Polydimethylsiloxane (PDMS) wrapped on the fabricated device. The standard β-Phase Perovskite polymer sensor with a sensing congregation can relay convergent sound directly to the nanofiber layer and the resultant sensitivity shows as high as 11 VPa⁻¹ at 40 Hz to 60 Hz incident frequency of normal conversation, which can be efficacious for developing high-performance acoustic based biomedical sensors.

1. Introduction
Implementation of an acoustic electro-magnetic wave, sound detection, in many domains like communication [1], medical science [2], health care sector [3], environmental safety [4], factory manufacturing [5], scientific research [6], defense application [7] and many more fields has been effectuated. Pioneering acoustic sensors ground on miscellaneous transducing principles such as piezoelectricity [8–10], piezo-resistance [11], piezo-capacitance [12] and piezo-optics [13] has been commenced. Piezoelectric polymer based acoustic sensors are resilient with high sensitivity and can be processed into wide geometries [14–16]. Nevertheless, almost all piezoelectric polymers are inevitably processed involving tedious method of mechanical extending and high voltage poling at an enhanced temperature to attain the piezoelectricity before making transducers [17], which are energy consuming and exorbitant cost. Recently, piezoelectricity of electrospinning nanofibers have been reported [18–21]. State-of-art stipulated that PVDF-TrFE co-polymer readily developed β-crystal [22–24] with exceptionally high capacitance in nano-Farad order at 30% trifluoroethylene (TrFE) molar ratio [25, 26]. This co-polymer is mundane for its Ferroelectric property that transfigures to paraelectric crystal with escalating temperature, otherwise Curie transition at 73 °C [27, 28]. Forthwith the developed piezoelectric polymer is contemplated as technically paramount polymer, having strong piezoelectricity without stretching and poling treatment [29–31] with massive piezoelectric coefficient and minimal dielectric loss [32, 33]. The co-polymers proffer advantages of outrageous flexibility and moderate acoustic impedance, begetting them competitive for various sophisticated applications such as ultrasound comprehension in organic medium [34, 35], energy ingathering from mechanical courses in nature [36, 37] and human movements as compared to piezoelectric ceramics [38]. The PVDF-TrFE nanofiber based sensors are using widely in various applications [6, 8, 13]. Piezoelectric nanofibers have shown great potential for application like mechanical sensing elements and energy generators [39]. The voltage outputs of inconstantly aligned nanofibers under compression or bending are more
2. Materials and experimental methods

2.1. Preparation of materials
Commercially available powder form of PVDF-TrFE (Sigma-Aldrich Chemical Co.) with molar ratio of 70:30 was dissolved into 12 wt% of methyl ethyl ketone (MEK) solvent. For complete dissolution of the powder into the solution, it was undergone through magnetic stirrer for three hours at 120 °C temperature. Quantitatively 1.8 gm of the powder was dissolved into 15 ml of MEK. The solution was sonicated for fifteen hours prior to electrospinning process. An automatic electrostatic based system of Bruker made was used to contrive nanofiber webs through Taylor cone formation and electrostatic repulsion at controlled atmospheric conditions. Prepared polymer solutions were placed into a 20 ml syringe with stainless steel needle (inner diameter: 0.85 mm) attachment. Electrically earthed up copper plate, enclosed in aluminum foil acted as a collector of the production unit located 14 cm at the fore front of the needle tip. Amidst the needle tip and the copper plate, high DC voltage of 8 KV was applied, while the infusion pace was set to 4 ml h⁻¹. The system took approximately four hours to complete the nanofiber production in closed condition at temperature of 32 °C under 49% humid condition. The electrospun nanofibers were then dried for few minutes to remove from collector. Finally, freestanding flexible films of congested nano webs on aluminum foil and on copper foil were achieved. Figure 1(a) shows the detail steps of solution preparation and figure 1(b) demonstrates needle based electrospinning of nanofibers. The entire method was repeated for PVDF-TrFE with molar ratio of 80:20 and 60:40 respectively.

2.2. Sensor fabrication hierarchy
Fabrication of sensor was followed by cutting of the nanofibers film just availed from the electrospinning into pieces with approximate dimension of 200 mm². The acoustic sensor was prepared by sandwiching a piece of nanofibers film with thin metallic foil having good electro-conducting property as electrode and transparent coating of PDMS (thickness 80 mm) membrane was made over the film. Aluminum was already present on one veneer of the nanofibers film which manoeuvres as an electrode of the sensor. On the other surface of the sensor, copper (Cu) foil was clamped on the film to perform as a second electrode. The metal facets were connected with the nanofiber layer, and province to accumulate electrical signals. Single gauge copper wires connected on both of the electrodes to transmit output response of the sensor. Two different type of sensors were fabricated (Aluminum-Aluminum electrode based sensor and Aluminum–Copper electrode based sensor) among them some were wrapped with fine layers of PDMS. Figure 1(c) represent physically obtained PDMS wrapped sensor and figure 1(d) indicates different layers of the fabricated sensor.

2.3. Material characterization

2.3.1. Morphological observations under scanning electron microscope (SEM)
Morphological analysis and surface properties of the nanofibers were examined under elevated resolution scanning electron microscope (Zeiss, FE-SEM). SEM images delineates about the surface morphology, particles homogeneity and external atomic arrangement of the co-polymer nanofibers web grown on the 0.1 mm thin aluminum foil. Pictographs were taken by FE-SEM at different accelerating voltages within the permissible range from 5 KV to 20 KV with alternative usable range of magnification from 500 X–50 KX for characterization of microscopic structures of the nanofibers sensor. Porous surface geometry of the nanofibers indicate TrFE doping within PVDF. No significant morphological variation was detected in VDF/TrFE ratio by SEM study of
the electrospun fibers. However, depending on concentration of the PVDF-TrFE solution, porosity of the fiber changes and the surface of some fiber represent smooth porosity, whereas other represent rough porosity and/or no porosity. Figure 2(a) and its corresponding expanded images shown no porosity on the film surface for overdiluted samples of PVDF-TrFE at molar ratio 60:40 whereas figure 2(b) and its corresponding expanded images represent smooth surface with some pores on the film surface of small in size for the same sample of PVDF-TrFE at optimum dilution.

Figures 2(c) and (d) with corresponding expanded images represent rough and smooth surfaces respectively having pores of comparably larger in size for samples of PVDF-TrFE pertaining molar ratio of 70:30 and 80:20 respectively.

2.3.2. Observation under x-ray diffraction (X-RD):
Through analysis of PVDF-TrFE nano-fibers film and PVDF film alone without any co-polymer doping obtained from electrospinning were undertaken by using standard x-ray diffraction (XRD, Rigaku 800, table top model) at 2\(\theta\) versus intensities with varying \(\theta\) values from 30\(^\circ\)–90\(^\circ\) and scanning speed of 4 rpm. Operating parameter of the x-ray generator was fixed at 40 KV and 40 mA. Two different XRD patterns of TrFE doped nano-fibers film and PVDF film alone along with their Gaussian profile and Full Width at Half Maxima (FWHM) of fabricated films are illustrated in the following figures 3(a) and (b).

2.3.3. Spectroscopic observation by Fourier Transform Infrared Reflection
PVDF-TrFE nano-fibers film was inserted into the particular position of the machine (Bruker Optics) to perform Fourier-transform infrared reflection spectroscopy (FTIR) in attenuated total reflection (ATR) mode within a range of 700–1500 cm\(^{-1}\) at resolution of 2 cm\(^{-1}\). Scanning of the film under FTIR was performed from 32 \(^\circ\)C initial temperature to 150 \(^\circ\)C maximum temperature and captured total 118 numbers of random data for entire heating cycle. Similar observation was also recorded for cooling of the film to its initial temperature reached at the time of heating. Figure 4(a) demonstrate FTIR graph for heating cycle of PVDF-TrFE nano-fibers film and figure 4(b) represents combined graph of the film and intersection points of the heating-cooling cycles.

2.3.4. Necessary arrangement for signal analysis
Signal analysis was performed through a special arrangement to facilitate conversion of divergent incident acoustic sound which is likely to be convergent electromagnetic beams, and impact on the nanofibers sensor directly through a hollow metallic cylinder with narrow hole at the sensor’s interface. Details of the arrangement demonstrate at figure 5. A sound system enable to emit sounds of different frequencies attached with a metallic cylinder was utilized to converge the sound source. One end of the metallic cylinder was closely attached with the
sensor device keeping no air gap in between. To avoid signal interference and external acoustic influence on the testing equipment, all other acoustic sources were kept in complete shutdown mode. The testing equipment and the sensor device were fixed in a rigid and firm condition with adjustable frame. In this way, the unwanted
influence from background, supporting base and total arrangement had negligible contribution to the signal output of the sensor device even at very low acoustic signals. The sensors were tested using a commercial PC suite speaker (Logitech) as a sound source. The sound frequency was controlled by a computer using in controller mode. The same PC suite base system with Lab view facility was used to view and record the output results.

3. Result and discussion

3.1. Structural analysis
The mole ratio of VDF/TrFE co-polymer and the annealing process are usually associated to constitute crystal lattice of PVDF-TrFE. The output response of the film were perceived an influential effect from orifices consists on film surface of the fabricated module. Sensitivity of the PVDF film alone without any co-polymer doping consist no pore on the surface which can’t rely within the reasonable level from incident acoustic signals. The output responses were distinguished by comparing both the module (SUPPLEMENTARY figure 1 is available online at stacks.iop.org/NANOX/1/020027/mmedia) from same acoustic incident signals demonstrate at
Figure 6. It is well known that chemical addition can change surface tension and viscosity of the effective solution. In the annealing process of nanofibers film formation, the effective force of adhesion by the TrFE molecules with the surface is higher than the force of cohesion by the analogous molecules which cause to form concave surface of the solution, hence, at dried state resultant adhesive forces tear up the film inward prevailing porous surface of the film and the porosity is changing with slight change in chemical concentration of TrFE. Therefore it was revealed that more the pores provide better acoustic response which implies higher concentration doping of TrFE with PVDF and formed more thick fibers. The incident can be elucidated by the effect of pores on sound propagation. Direct exposure of convergent acoustic waves on nanofibers produce higher electrical output. Thickness of the nanofibers web also affected the output response. When lamina of the nanofibers film increased or wrapped with PDMS, the device sensitivity is increased significantly (supplementary figure 2). The electrode materials shown culmination on the output voltage and sensitivity. Use of two different materials as electrode
Table 1. The comparison of sensing activity of fabricated PVDF-TrFE sensors.

| SL No. | Sensor type  | Type of input signal  | Input signal in (Hz) | Input signal in (dB) | Sensitivity (S) in (VPa⁻¹) | % error in S* |
|--------|--------------|-----------------------|----------------------|----------------------|-----------------------------|---------------|
| 1      | Al-Al electrode without PDMS wrapped | Normal conversation | 40–60                | 60                   | 1.9                         | 5.26          |
| 2      | Al-Al electrode with PDMS wrapped   |                       |                      |                      | 6.5                         | 1.53          |
| 3      | Al-Cu electrode without PDMS wrapped| Soft music            | 440                  | 80                   | 7.5                         | 1.33          |
| 4      | Al-Cu electrode with PDMS wrapped   | Rock music             | 75                   | 105                  | 10.5                        | 0.95          |
| 5      |                             | Slow sound             | 25                   | 0                    | 1.0                         | 10.00         |
| 6      |                             | Loud sound             | 15500                | 120                  | 0.4                         | 0.25          |
| 7      |                             | Cardiac sound          | 0.05–120             | 0                    | 0.1                         | 1.00          |
| 8      | Mechanical sound              | 8000                  | 85                   |                      | 3.5                         | 2.80          |

* (Method of dynamic resistance measurement and percentage (%) error calculation are mentioned at supplementary information 9 and 10 respectively).
furnish better sensitivity over same type of electrode (table 1) as potential difference between two individual electrodes provide some initial potential to the sensing system which ultimately causes higher throughput to the sensor. The output voltage and sensitivity of the sensor shown a linear increment by using thin cover of PDMS grown up on the sensor (table 1) when other parameters remain unchanged, indicating that the material also contribute to convert sound waves into electricity by polarizing electrical charges towards the surface of the sensor. A meticulous perception on culmination of the doping concentration of TrFE are shown in figures 2(a) to (d) and the acoustic to electric conversion aptness of nanofibers web with different pore diameter may vary, which can be reported in further future conveyance. The x-ray diffraction (XRD) pattern and Fourier transform infrared (FTIR) spectrum indicate that the nanofibers mainly contain α and β crystal phases. XRD measurement demonstrated in figure 3(a) indicates about emergence of Ferroelectric β phase of the films. XRD pattern of PVDF alone in figure 3(b) clearly distinguishes the enhancement in crystal characteristics with TrFE doping. FTIR spectra of the PVDF-TrFE films for heating cycle in figure 3(a) and combined FTIR spectra in figure 3(b) for heating-cooling cycle confirmed some Ferroelectric properties of the TrFE doped film. The right handed swipe from the intersection points (77, 0.938) precisely represent a Ferroelectric loop. Temperature of the intersection point was very closer to the Curie transition point at 73 °C which indicates para-electric transformation property of the material. The asymmetry between two phases (heating-cooling) in combined FTIR spectra provides some useful information about the presence of functional groups which make the film intrinsically charged. It was also an indication about the changing property of the film with respect to thermal change.

3.2. Analysis of surface morphology
As a whole, the experimental analysis of surface morphology of the film were performed based on the arbitrarily orientated nano fibers web having fiber diameter of 250 ± 30 nm (shown in the histogram of diameter distribution in supplementary figure 3). It was anticipated that the fiber density and orientation will affect the signal conversion efficiency. Consequentual interpretation of fiber density was observed for randomly oriented fibers web prepared in different film thickness. The fibers attained higher thickness shown extended number of pores, while others represented minimum pores, concluded that fiber thickness indeed influences sensor behavior. All the fabricated films shown acoustic to electricity conversion potential with varying piezo-potential. Under the audible sound frequency, thick fibers film achieved better response than thin fibers film (supplementary figure 4). The output response of the sensor was revealed from symmetric and asymmetric electrodes used for fabrication, PDMS wrapped, unwrapped and various incident acoustic signals were noted in table 1. Farther, arbitrarily orientated PVDF-TrFE fibers web, nanofibers film of PVDF alone without any co-polymer doping were also prepared for reference. In a particular film thickness, nanofibers film made of PVDF alone without any co-polymer doping shown no pores on the surface and displayed low response (see in Supplementary figure 5) in comparison with PVDF-TrFE film. On one side of the surface, ions with opposite polarity formed, if an incident signal was applied to the sensor, electrons were pushed and protons were pulled from the positive and negative end of the device. Oppositely charged ions of the electrodes were separated by creating positive and negative charge affinity to the surface of the electrode and provided effective potential difference for electrical activity of the device. The amount of potential difference was dependents on input signal of the device. Previous studies (see 1–5 of supplementary information) revealed that, PDMS acts as a dielectric material and well suited for capacitive sensing applications like pressure sensor, flexible electric circuits and biosensors to measure bio-potential such as EEG, ECG, and EMG. Based on the data provided by the manufacturer, varying dielectric constant of PDMS is considered within the range from 2.68 up to 2.72 depending on the application frequency of the desired electrical field. Therefore, PDMS wrapping enhance overall electrical conductivity of the fabricated sensor which ultimately increase the effective output performance of the sensor.

3.3. Sensing activity of the device
Output electric signals were generated due to compound conductive properties of the sensor from different incident acoustic sound waves as described in figures 6(a)–(d), descend on the nanofibers device. Incident signal generates an instant pulse to the electron, proton and conductive ions that exist on the surface of the sensor which vehemence to polarize electrical charges and mainspring potential difference between the two electrodes. The high content of β crystal phase and orientation of macromolecular chain dipoles expedites piezoelectric conversion under compressive impact towards the nanofibers film thickness. The device generated a periodic voltage output within the peak value ranging from 3.50 V to 11.50 V at the incident sound wave of frequency ranging from 30 Hz to 15 KHz for different sounds like normal conversation, sweet melody, rock music, whispering and telephonic conversation (see in table 1). The output response was like a typical alternating current.
Fast Fourier transform (FFT) and Short Time Fourier transform (STFT) signal processing technique were utilized to process the output signals and to minimize the noise interference from the background sound in the testing environment, electronic inter mediator and connecting wires. The output voltage were analyzed by keeping the sound pressure fixed while varying their frequencies which was affected by acoustic wave frequency. As proclaimed in figure 6, the voltage output, signal amplitude, phases and color distribution of the signal throughput changes abruptly with respect to incident signal frequencies. The maximum voltage output of 11.50 V was obtained at 3 KHz incident acoustic signal (supplementary figure 6). When the frequency of the incident wave was above 15 KHz or below 30 Hz, a very low output response was displayed (figure 6(d2) and in table 1). In this range the acoustic output ceased nearly to zero. Signal fluctuation in the output was observed for rock music of 400 Hz –1,500 Hz represented in figure 6(b2). Schematic diagram in figure 6(a2), proclaimed for normal conversation within 40 Hz to 100 Hz resulted nearly stable output response of 9.50 V. The output response of average 5.0 V was obtained from soft music of 440 Hz, illustrated in figure 6(c2). Sensitivity is a pivotal parameter to indicate feasibility of the sensor. The sensitivity (S) for acoustic sensor can be calculated by following equation (1) [1, 35]:

\[ S = \frac{V}{P} = \frac{V}{P_0}10^{\frac{L}{20}}. \] (1)

Where, \( P \) is the sound pressure, \( V \) is the voltage output of the device, \( P_0 \) is the reference sound pressure of 0.00002 Pa and \( L \) is the SPL in decibels. In this experiment keeping \( P \) unchanged, sensitivity of the fabricated device become equivalent to output voltage response. Most interestingly using this sensor cardiac response within the frequency range of 0.05 Hz – 120 Hz at 0 dB was detected with a sensitivity of 10 mV and the signal peak clearly represented QRS complex of the conventional ECG analogous (table 1 and supplementary figure 7). Output characteristic was also evaluated by applying mobile vibration, hand sewing machine, finger tapping and hand punching (supplementary figure 8) for further comparison. It is interesting to mention that the device performance was outstanding within the audible frequency range. Table 2 represented a comprehensive comparison between the obtained results in our experiment and reported study from already published literature.

4. Discussion

Electro spinning based PVDF-TrFE film demonstrated higher crystallinity because of electro spinning stretching through the fabrication process. TrFE doping provide porous surface structure of the film, make it suitable as a acoustic sensor. Combined graph of heating-cooling cycle at figure 4(b) represent asymmetry between two phases (heating-cooling) in FTIR spectra may be due to rapid fall of temperature in cooling cycle. Though the actual reason behind such a variation is not clear and yet to be investigated which may reveal interesting properties of polymers. In figure 6, response due to various acoustic signals represent better performance for normal conversation at 40–60 Hz acoustic frequency. Even it can displayed sensitivity of 10 mV for very sophisticated cardiac pulse of frequency range 0.05 Hz – 120 Hz at 0 dB and the signal peak clearly represented QRS complex of the conventional ECG analogous at supplementary figure 7. Performance of the fabricated sensor for diverse applications were summarized in tables 1 and 2. The PDMS coated PVDF-TrFE sensors shown better performance compared to the sensors not coated with the same. The maximum output voltage of 11.50 V was obtained at 3KHz frequency of incident acoustic signal shown in figures 6(a), (b) and supplementary figure 6, which also observed in the frequency range of normal conversations. Therefore, the fabricated sensor is capable of providing a vast range of applications. As the response from the fabricated sensor is uniform throughout the application frequencies, low drift appeared for the sensors. The metal electrodes of the sensor displayed better response even at asymmetric mechanical incident signal like mobile vibration, stress from small hand sewing machine, finger tapping and hand punching. In fact, the PVDF-TrFE nanofibers are flexible and they are facile to vibrate under acoustic incident. PDMS films shown very interesting mechanical characteristic which comprises massive PDMS bulks are homogeneous, isotropic and highly deformable. However, device vibration caused by acoustic absorption is habitually localized, and deformations of sensor caused by repeated constriction or contorting of fibrous web within the assembly by virtue of the mechanical deformation in the spans of whole fibrous structure. Under acoustic signal, the PDMS on the device also vibrates and thus intensifying the overall vibration of the film near to the covered areas. Under acoustic signals, the nanofibers web within the device could vibrate in all the probable directions within the occupied plane. The directly exposed nanofibers vibrate more intensively than those covered with the PDMS. Such asymmetry in fibre vibration enhances the chance of overall deformation.
| Sl No. | Sensor type | Applied input signal | Method of fabrication | Dynamic resistance | Output voltage | References |
|-------|-------------|----------------------|-----------------------|-------------------|---------------|------------|
| 1     | Al-Al electrode without PDMS wrapped | 40–60 Hz | Electrospinning | 2.0 MΩ | 1.9 V | table 1 |
| 2     | Al-Al electrode with PDMS wrapped | 440 Hz | 2.5 MΩ | 6.5 V |
| 3     | Al-Cu electrode without PDMS wrapped | 75 Hz | 1.8 MΩ | 2.1 V | |
| 4     | Al-Cu electrode with PDMS wrapped | 25 Hz | 3.2 MΩ | 9.5 V |
| 5     | Al-Cu electrode with PDMS wrapped | 15500 Hz | | 7.5 V | |
| 6     | Al-Cu electrode with PDMS wrapped | 0.05–120 Hz | | 1.0 V | |
| 7     | Al-Cu electrode with PDMS wrapped | 8000 Hz | | 0.4 V | |
| 8     | Graphene/P(VDF-TrFE)/graphene multilayer film | 1.0–3000 Hz | Acoustic actuator and nano generator | 450 Ω | 3.0 V | [6] |
| 9     | Strain sensor | 34 mm/s (at 5 Hz) | Far-field electrospinning | No | 2.21 V | [7] |
| 10    | Pressure sensor | 0–300 mm Hg | Wafer based flexible sandwich sensors | No | 99 μV | [13] |
| 11    | Aluminum foil electrode | 100 Hz | Electrosprin deposition | No | 0.78 V | [14] |
| 12    | Graphene electrode | 4 Hz | Electrosprin deposition | 1392 Ω/sq of sheet | 2.5 V | [19] |
| 13    | Piezoelectric PVDF nanogenerator | Strain of 2 Hz | Near-field electrospinning | 15 GΩ | 5–30 mV | [20] |
| 14    | Hybrid Nano generator | Particular strain fixed and applied over 0.06, 0.04, and 0.03 s. | Electrosprin-based piezoelectric poly(vinylidene fluoride) nanofiber nano generator for harvesting mechanical energy | 10 MΩ | 15 to 20 mV | [21] |
| 15    | P(VDF-HFP) micro-porous electro-active films | Repeated finger touch motion | Hydrated salt filler assisted sponge like flexible piezoelectric generator (FPG) | No | 8.0 V | [30] |
| 16    | Porous PVDF film | Variable pressure | Pressure sensor | No | 3.4 V | [33] |
| 17    | contact vibration sensor | 16 Hz to 3 kHz | PVDF film bounded with rubber | No | 0.3 mV | [35] |
5. Conclusion

In the commenced work, piezoelectric based PVDF-TrFE nanofibers consisting of acoustic to electric energy transformation property was successfully demonstrated. The high output response at 3 KHz frequency coincides within the medically recommended most sensitive frequency range for human ear. The sensor device offers better sensitivity than piezoelectric PVDF film and provide excellent ability to differentiate different sound waves in the low to medium frequency region. Since the device shows higher sensitivity at 3 KHz and within the discernible frequency range, it is especially useful for audio metric device fabrication. In future, the sensor might be useful for developing various acoustic sensors for novel application.

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Data availability

Our data can be available on request.

Competing interests

The authors declared no competing interests.

Authors’ contributions

F K and S N P designed the study, F K and W R prepared all samples for analysis. F K and W R collected and analyzed the data. F K and S N P interpreted the results and wrote the manuscript. All authors gave final approval for publication.

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Research ethics

The research did no present any ethical considerations. Ethical approval were not required to complete an ethical assessment prior to conducting this research.

Animal ethics

The heading does not apply.

Permission to carry out fieldwork

The heading does not apply.

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