Long-term power degradation testing of piezoelectric vibration energy harvesters for low-frequency applications

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Abstract
Piezoelectric energy harvesters represent a viable and well-proven solution to convert ambient vibrations into useful electric power within a number of modern life applications. Whilst a large amount of studies has focused on improving power output from these devices, relatively little research has been directed to investigate how these devices degrade over time and the effect this has on long-term power generation. This paper, therefore, aims to experimentally investigate how piezoelectric vibration energy harvesters degrade during long-term operation in realistic harvesting conditions. The harvesters tested are unimorph cantilevers based on three of the most commonly used piezoelectric options: polyvinylidene fluoride (PVDF), Macro Fiber Composite (MFC), and Quick Pack (QP). Testing was carried out under single-frequency excitation (10–40 Hz) of 1g amplitude for three million vibration cycles. Our results show that the natural frequency and the optimum load resistance of the harvesters may vary during prolonged operation. Importantly, a larger cumulative variation in natural frequency and optimum load resistance yields a larger variation in power output, thereby linking the variation in power to the variation of the mechanical and/or electrical properties of the harvesters. Comparing the average power values over the testing period we found that increasing the tip mass does not necessarily improve the average power output, suggesting that a larger tip mass may exacerbate the degradation of the mechanical and/or electrical properties of the harvester. This was particularly evident for the stiffest QP harvesters which showed the highest signs of power degradation; nevertheless, QP harvesters still managed to demonstrate the highest power density values. When cost consideration is taken into account in the assessment, PVDF harvesters managed to demonstrate the highest power density to cost ratio.

1. Introduction

Wireless sensor networks comprise a group of spatially dispersed and autonomous sensor nodes, varying in number from a few to several hundreds or even thousands, that monitor physical conditions of the environment by measuring parameters such as temperature, pressure, humidity, sound level, light intensity, chemical concentration, wind speed, among several others. Besides being an enabling technology for the Internet of things [1], wireless sensor networks are presently used across a broad range of applications such as industrial process monitoring [2], damage detection in machines and structures [3], quality monitoring of air [4] and water [5], wildfire detection [6], and natural disaster prevention [7]. The sensor nodes that comprise a wireless sensor network typically include, in addition to the sensor itself, a basic processing and radio transceiver unit and a power source, this latter usually is in the form of a battery.

When the sensor network comprises a very large number of sensor nodes, or when the sensors are intended for mobile applications or for deploy-and-forget operation in remote or harsh environments, the periodic
replacement of the batteries may become challenging or impractical. In these cases, batteries can be replaced with energy harvesting modules which convert ambient energy into electric power [8–12]. Energies that can be harvested from the environment include kinetic energy from wind [13–15], waves [16], water flows [17–19], ambient vibration [20–22], thermal energy in the form of temperature variations in space or time [23–25], solar radiant energy [26], electromagnetic energy from radio and television broadcasting [27], chemical energy from salinity or other concentration gradients [28], biomechanical energy [29, 30], and combinations thereof [31–35]. Due to the ubiquitous presence of ambient vibrations, considerable attention has been devoted to the conversion of mechanical vibration energy into electric power exploiting electromagnetic/electrostatic induction or piezoelectricity [21, 36, 37]. The piezoelectric effect provides higher energy density and is therefore normally preferred for harvesting ambient vibrations [38].

Piezoelectric vibration energy harvesters (PVEHs) are normally designed with a thin and flat geometric shape to promptly react to the motion of the host structure, such as the cantilever beam geometry which is the most frequently used structural design for this type of harvesters [21, 38]. The choice of the piezoelectric material, ceramics (such as PZT-lead zirconate titanate) or polymers (such as PVDF), is essentially determined by the frequency of the ambient vibration of interest, which typically vary within 1–200 Hz [39]. Being rigid, harvesters based on piezoelectric ceramics have high resonant frequencies, and are therefore suitable for high frequency applications (indicatively, 50–100 Hz or higher). On the other hand, harvesters based on piezoelectric polymers are typically of high flexibility and have low resonant frequencies, and are therefore more suitable for low frequency applications (indicatively, up to 50–100 Hz) [39].

The technology of PVEHs has gradually matured through the extensive research carried out in the last decade [40–42], and the successful self-powering of sensors and small electronic devices using PVEHs has been repeatedly demonstrated [38]. Whilst current research is looking into design optimization and performance enhancement [43–46], one key aspect that has seemingly been overlooked is the long-term stability of PVEHs. As a consequence of the laws of physics, and notably of the second law of thermodynamics, wear and tear naturally and inevitably occur in any product as a result of normal aging, thus limiting the product service life. As a necessary step in product development before commercialization, endurance testing is routinely performed to gauge the capacity of a product to last or withstand wear and tear and provide evidence of how its performance varies with time, thereby assessing the durability and performance stability of the product over prolonged operation in representative operating conditions. PVEHs are electromechanical systems that generate electric power by inducing a cyclic stress in the piezoelectric material using the ambient vibration. Any degradation in PVEH performance can therefore be attributed to a degradation of the mechanical properties (stiffness and/or damping), a degradation of the electrical properties (impedance), or a combination of both [47]. With PVEHs of cantilever beam geometry, in particular, the induced stress is concentrated near the fixed root of the beam. This location is where mechanical degradation would be expected to occur from stress-induced fatigue due to the cyclical nature of the loading. More often than not, the resonant frequency of the cantilever beam structure is higher than the frequency of the external exciting vibration, and a proof mass is usually added on the tip end of the beam so that the resonant frequency can be reduced to match the input frequency of the host structure [38]. Clearly, the inclusion of a proof mass can play a role in the durability and performance stability of the device.

Degradation tests on piezoelectric materials have been carried out in a number of studies for different PZT materials experiencing different loading conditions. For example, Tai and Kim [48] studied the degradation of piezoelectric properties under compressive cyclic loading as well as mechanical fatigue behaviours for Pb(Zr,Ti) 03 ceramics of tetragonal, morphotropic phase boundary, and rhombohedral compositions. They showed that fatigue resistance was highest for the rhombohedral composition, and that fatigue resistance could be lowered for all three compositions using poling treatment. In another comprehensive study, Cain et al [49] investigated candidates having the shapes of flat discs and long cylinders. The study dealt with simple uniaxial mechanical cycling of monolithic materials parallel to the polar direction under short circuit conditions. Electrical stressing has also been investigated. It has been shown that for repeated mechanical loading, soft materials are very sensitive with the softer composition PC 5H having a greater rate of degradation compared to PZT 5A when stress is increased. Hard materials showed less degradation due to mechanical cycling but were sensitive to extended operation under constant load. Cyclic electrical stressing results showed that soft materials are again more sensitive compared to hard materials; however, degradation due to electrical stressing was of less significance compared to that due to moderate mechanical cyclic loading. Elahi et al [50] also examined degradation of rectangular PZT-5A4E patches over a range of conditions for temperature (20 °C to 180 °C), load resistance (0 Ohm to 90 Ohm), and frequency (50 Hz to 250 Hz). They investigated up to 2500 stress cycles demonstrating that after 1500 cycles, electromechanical properties showed signs of degradation.

Some documented endurance tests carried out for vibration piezoelectric beams are also present. Pillatsch et al [51] studied degradation effects of a bimorph cantilever bending beam with tip mass whilst excited at the tip using magnetic actuation under symmetrical and asymmetrical sinusoidal loading. Besides loss of power, they demonstrated that extended use causes significant changes in the beam’s material properties including both
mechanical (stiffness) and electrical (impedance) properties. The change in stiffness would in turn affect the natural frequency of the device and a change in impedance would change the ideal load resistance of a circuit for maximum power. This result is consistent with a study by Selten et al.[52] who found that, in the case of mechanical compressive tests, the Young’s modulus and piezoelectric coefficients are strongly affected by loading history. Note that micro-cracks in the piezoelectric ceramic layer have been found in a number of studies, e.g. [51, 53, 54], and formation of such micro-cracks was typically considered a potential leading cause of degradation. However, possible fabrication solutions such as pre-stressing the material and laser lift-off[55, 56] could decrease degradation effects and enhance the strain capability of the piezoelectric material. In a recent study, Wang et al.[57] considered testing of beams with bonded MFC patches. They showed that stress depolarization can cause actuation function degradation of the piezoelectric devices ultimately causing the failure of the structural system.

There have also been studies comparing the energy harvesting performance of different devices. Sodano et al.[58] investigated three harvesting devices made from monolithic piezoceramic PZT, bimorph QP, and MFC. They recharged batteries with different capacities using these devices whilst measuring the charge time and maximum capacity battery that can be charged. It was found that QP and the monolithic piezoceramic PZT were capable of recharging the batteries with the latter being more effective within random vibration environments usually experienced with ambient vibrations. Later, Sodano et al.[59] compared MFC, QP IDE (model QP10ni), and QP (model QP10m) by attaching the three candidates to a cantilever beam and exciting it at twelve resonant frequencies. They used power density as a metric for comparison showing the superiority of QP compared to the two other candidates.

Overall, experimental studies have shown that piezoelectric devices do show degradation under different loading conditions. The majority of the available investigations has considered cyclic compressive loading, and comparatively fewer studies have considered cyclic bending loading. There are also some studies comparing energy harvesting performance of a number of piezoelectric material options. However, the performance degradation over prolonged operations of different piezoelectric harvesters has not been compared. Piezoelectric polymers are known for higher flexibility, environmental compatibility, and resistance to mechanical shocks than piezoelectric ceramics[35]. These qualities show promise in energy harvesting applications at low frequencies; however, to the best of our knowledge their performance degradation during prolonged operation has not been assessed. Finally, PVEHs typically employ a tip mass to lower the device resonant frequency to the required harvesting frequency as well as to increase the harvested power[35]. However, there is currently no systematic study that considers the tip mass effect on the harvesting performance over prolonged operations. Because of the aforementioned limitations, in this study the variation in performance during prolonged operation of three of the most frequently used piezoelectric options (PVDF, MFC, and QP) will be thoroughly investigated within the context of PVEHs for low frequency applications. To the best of our knowledge, no previous study has compared the performance degradation of these alternatives when employed as PVEHs in bending mode, creating the motivation for this study. As such, in this paper we analyse how the power output from these piezoelectric energy harvesting devices changes over time whilst shedding more light on the effect that the inclusion of a tip mass has on performance degradation.

The rest of the paper is organised as follows: section 2 introduces the built harvesters, the experimental setup, the test conditions, and the data processing approach. Section 3 provides results for the investigation of changing tip mass on degradation performance. It also shows results for how tuning the input frequency signal to match the changing device natural frequency could affect performance. Section 4 presents final concluding remarks from the study.

2. Materials and methods

2.1. Harvesting materials and configurations

The construction of the active and passive layers of the harvesting devices employed in this study are schematically presented in figure 1. Here, we considered three off-the-shelf piezoelectric options: PVDF, MFC, and QP. Even though these piezoelectric alternatives are significantly different in terms of mechanical and electrical properties, they are all designed for energy harvesting, and are therefore comparable in this respect. The first harvester built is based on polyvinylidene fluoride (PVDF) provided from TE Connectivity (www.te.com; model: LD72-028K/1; density: $\rho = 1780$ kg m$^{-3}$; Young’s modulus: $Y = 2.3$ GPa; piezo strain constant: $d_{31} = 23 \times 10^{-12}$ C N$^{-1}$; capacitance: $C = 2.85$ nF). The second harvester built is based on Macro Fiber Composite (MFC) provided from Smart Material Corp. (www.smart-material.com; model: M5628-P2; density: $\rho = 5440$ kg m$^{-3}$; Young’s modulus: $Y = 15.86$ GPa; piezo strain constant: $d_{31} = -170 \times 10^{-12}$ C N$^{-1}$; capacitance: $C = 113$ nF). The third harvester built is based on lead zirconate titanate PZT-5i wafer QuickPack$^\text{TM}$ (QP) from Mide Technology Corp. (www.mide.com; model: S128-J1FR-1808YB; density: $\rho = 9120$ kg m$^{-3}$; Young’s modulus: $Y = 81.2$ GPa; piezo strain constant: $d_{31} = -320 \times 10^{-12}$ C N$^{-1}$; capacitance: $C = 50$ nF). The construction of the active and passive layers of the harvesting devices employed in this study are
\[ \rho = 7800 \text{ kg m}^{-3}; \] Young’s modulus: \( Y = 51 \text{ GPa}; \) piezo strain constant: \( d_{31} = -210 \times 10^{-12} \text{ CN}^{-1}; \) capacitance: \( C = 100 \text{ nF}; \) PVDF is a highly flexible piezo-polymer, MFC is a moderately flexible piezo-ceramic, whereas QP is a relatively rigid piezo-ceramic. This difference in rigidity between MFC and QP is in that MFC is composed of piezoelectric fibers whereas QP is composed of piezoelectric sheets \([39, 59]\). PVDF has silver ink screen printed electrodes, MFC has interdigitated copper-tin electrodes patterned on polyimide film, and QP has copper electrodes. There is quite a recognisable difference in the mechanical properties of the three materials with PVDF having the lowest density and Young’s modulus whereas QP has the highest density and Young’s Modulus. This translates to a difference in rigidity giving PVDF the potential to realise harvesting devices for lower frequency applications (due to the expected lower natural frequency), and vice versa with respect to QP harvesters. Additionally, the electric properties of the three materials are quite different with MFC and QP having much higher values for capacitance and piezo strain constant in 3-1 mode. This leads to differences in the ability to generate electric power when exposed to vibration in the 3-1 mode. It is worth noting, however, that current research is looking into improving the electrical properties and performance of PVDFs \([60]\).

The QP device used as part of these tests comes readily assembled from the manufacturer and involves one piezo layer. As such, we only employed one layer of MFC and PVDF when manufacturing these harvesting devices to ensure some sort of consistency. Because QP has relatively higher stiffness there was no need to further stiffen it with a passive elastic layer. On the other hand, both MFC and PVDF required stiffening to allow meaningful structures. Hence, both MFC and PVDF were bonded to a passive elastic layer made of stainless steel provided by Precision Brand (precisionbrand.com); density: \( \rho_e = 7900 \text{ kg m}^{-3}; \) Young’s modulus: \( Y_e = 180 \text{ GPa}; \)
thickness: \( h_e = 0.1 \text{ mm} \). The elastic layer was attached to both the MFC and PVDF using a very thin layer of epoxy of negligible thickness but sufficient to ensure effective bonding. As shown in figure 1, the final configuration for the MFC and PVDF harvesters is essentially a unimorph configuration (one active piezo layer and one passive elastic layer), a configuration widely employed in piezoelectric devices [36, 37]. On the other hand, the QP harvester has also one active piezo layer but wrapped in a passive epoxy resin, and no added passive elastic layer. Note that epoxy is not equally distributed on both sides of the active layer. In fact, there is more epoxy to one side than the other allowing the QP harvester to have strong resemblance to a unimorph configuration. Even though the QP harvester is not, strictly speaking, of unimorph design, all harvesters considered here have a similar configuration (i.e. one active piezo layer and a passive substrate), similar enough to allow a meaningful comparison.

The developed devices were clamped in a custom-made laser-cut acrylic housing (see figure 2) to allow rigid attachment to the shaking device. Note that the portion of the harvester enclosed within the clamp is strictly fixed, does not deform, and therefore does not produce any power. To investigate the effect of tip mass inclusion, set amounts of mass were added to the tip of each device. The value of tip mass to be attached was evaluated based on the required ratio of the tip mass to the equivalent mass of the harvester beam. The equivalent mass of the harvester beam was evaluated based on the Rayleigh–Ritz approach in which the total mass of the cantilever beam is scaled by a factor of 33/140. This is essentially an idealisation of the harvester beam as a mass/spring system that allows easy representation/modulation of the device natural frequency due to the addition of a tip mass. In fact, this method is well adopted by manufacturers such as Mide Technology Corp. (manufacturers for QPs) who provide applicable data up to a tip mass ratio of 50 [61]. In this study, tip mass ratios of 10, 20 and 30 were chosen to cover a range of values that may be used in realistic operations.

Figure 2. Harvesting devices attached to a custom-made acrylic clamp. (a) Devices with no tip mass; (b) and (c) example devices with tip mass set at different angles for better visualisation.
Metallic tip masses with the required weight were firmly attached to both sides of the device in a symmetric fashion. Each tip mass was ensured to extend along the entire width of the beam. The centre line of each tip mass (on each side) was set to be coincident with the tip edge of the beam. High-strength adhesive tape was used to ensure tip masses fixation during testing. Figure 2 shows a selection of devices with and without affixed tip masses.

2.2. Experimental setup

Figure 3 shows the experimental setup used during testing. A signal generator (by Tektronix, model AFG1022) was used to produce a sinusoidal signal sent to the shaker (by Data Physics, model V55) via an amplifier (by Data Physics, model PA300E). Each energy harvester tested was firmly fixed to the shaker unit and was excited at its base. An accelerometer (by PCB Piezotronics, model PCB 336M13) was fixed close to the base excitation point of the harvesters to monitor the vibrational acceleration where this signal was amplified and then measured. Each harvesting device was connected to its corresponding optimum load resistance value. A thermocouple (by RS Components, model RS PRO Type K) was used to monitor the ambient temperature in the testing environment. The temperature and accelerometer readings as well as the voltage across the resistance were all recorded using a Data Acquisition unit (by National Instruments, model NI-USB-6225). This unit in turn fed the values to a computer running LabVIEW 2017 for real-time results monitoring and recording. The power generated by the harvesters was calculated in LabVIEW as the product of the voltage measured across the load resistor times the current flowing through the load resistor. Finally, a cooler unit was used to avoid overheating the shaker unit during the long operational times.

2.3. Testing conditions

The conditions for testing were designed to match, as much as possible, realistic scenarios. It is well documented that a frequency range of up to 200 Hz with an acceleration up to 1.5 g represent the operational range for most sources of ambient vibration [39]. Without some sort of active control, energy harvesting devices are typically designed for operation at targeted frequencies. Indeed, PVDF, MFC, and QP harvesters each have different mechanical properties making them suitable for harvesting energy at different frequencies. With no tip mass, the length dimension was used to tune the first natural frequency at 50, 110, and 150 Hz, for the PVDF, MFC, and QP harvesters, respectively. Note that, the PVDF harvester is designed for the lowest frequency because it has the lowest stiffness (highest flexibility), whereas the QP harvester is designed for the highest frequency because it has the highest stiffness (lowest flexibility). The values of 50, 110, and 150 Hz represent good baseline values which, with the addition of tip masses, will be lowered to 10–40 Hz: a range representative of low-frequency ambient vibrations. Table 1 shows the dimensions of the realised harvesting devices for testing. As explained in section 2.1, tip masses were varied to achieve tip mass ratios of 10, 20, and 30. To produce a meaningful and easy to follow designation for each test case, we name our tests as Material–Tip Mass Ratio. For example, PVDF–20 thus indicates the test case for the harvester comprising PVDF and having a tip mass ratio of 20.

We decided to keep the base excitation acceleration level constant at 1 g during the tests. Two reasons were behind this selection: firstly, it is a representative value for typical excitation levels from ambient vibrations [39]; and secondly, this allows all measured quantities to be regarded as per unit g excitation. Table 2 shows the standard deviation values of the excitation level from the target mean 1 g value for the tests conducted. The mean and maximum standard deviation values for the nine tests is 1.5% and 4.7%, respectively. These values confirm that the excitation acceleration remained constant at 1 g to within a few percent throughout the tests.

Figure 3. Experimental Setup used in the current study.
Table 1. Geometric characteristics of the developed harvesting devices to achieve desired natural frequency.

| Device (including passive and active layers) | Length (mm) | Width (mm) | Thickness (mm) | Volume (cm$^3$) | Natural frequency (Hz) |
|---------------------------------------------|-------------|------------|----------------|-----------------|----------------------|
| PVDF MFC QP | 52 46 47 | 17 32 26 | 0.42 0.52 0.69 | 0.38 0.75 0.82 | (without tip mass) |
| Piezo Element (active layer only) | Length (mm) | Width (mm) | Thickness (mm) | Volume (cm$^3$) | Natural frequency (Hz) |
| PVDF MFC QP | 50 45 39 | 13 28 21 | 0.040 0.30 0.69 | 0.027 0.38 0.56 | (without tip mass) |

Table 2. Testing conditions for the current experiment.

| Harvester | Excitation standard deviation (%) | Natural frequency (Hz) | Optimum load resistance (kOhm) | Total run time (h) |
|-----------|-----------------------------------|------------------------|-------------------------------|-------------------|
| PVDF-10   | 0.32                             | 17                     | 2300                          | 49                |
| PVDF-20   | 0.52                             | 11                     | 1700                          | 76                |
| PVDF-30   | 4.7                              | 10                     | 2000                          | 83                |
| MFC-10    | 0.61                             | 24                     | 34                            | 35                |
| MFC-20    | 0.76                             | 20                     | 37                            | 42                |
| MFC-30    | 0.31                             | 12                     | 43                            | 69                |
| QP-10     | 0.59                             | 40                     | 11                            | 21                |
| QP-20     | 1.2                              | 30                     | 12                            | 28                |
| QP-30     | 4.4                              | 25                     | 15                            | 33                |

Table 2 provides the settings for the set of tests conducted. In this table, the value of the optimum resistance that would allow the maximum power generation is provided. This value was empirically determined through conducting an experimental power scan with different resistance values, and identifying the optimum resistance value that would allow maximum power generation. At the start of each test the natural frequency and optimum load resistance of each harvester were measured. As discussed later, this was repeated after the testing was concluded to assess any variation of the natural frequency and optimum load resistance that may have arisen from change in device properties due to prolonged operation. Note that the natural frequency and optimum load resistance values included in table 2 are those measured at the start of the tests. Finally, it is important to stress that, for each test case, a brand new piezoelectric layer/material was used to ensure no previous hysteresis, memory, or fatigue effects were present.

The testing of the harvesters was performed under single-frequency excitation. At the beginning of each test the excitation frequency of the shaker was tuned to the resonant frequency of the harvester in table 2, and was not changed afterwards. To thoroughly examine long-term effects, instead of running the tests for a specific length of time, the tests were run for a predefined number of vibrational cycles to allow better comparison between devices. Tests were run for three million cycles representing a good compromise between being sufficient timing to identify if degradation effects exist and allowing good use of testing time. Note that this represents a significant increase of testing times compared to some of the previous studies that conducted tests on the order of thousand cycles, e.g. [50]. Table 2 provides the corresponding total run time for each test case. As can be seen, the PVFD-30 test case required the longest testing time of 3.5 days, whilst the testing of the harvesters QP-10 lasted for 21 h. The total cumulative testing time for all harvesters was 436 h, which corresponds to around 18.2 continuous operation days.

As noted by Kim et al [62], a change in ambient temperature can cause a change in power output of a piezoelectric device. In particular, they found that the power output from PZT decreased with increasing
temperature, and that the temperature effect becomes most impactful when over 40 °C approximately. The ambient temperature was recorded during the present tests, and the results are provided in table 3.

As can be seen, the ambient temperature during each test remained constant to within 1.2 °C. Any effects of the operating temperature on the present harvesters’ performance, therefore, should be minimal and negligible.

2.4. Data processing
The power and acceleration signals from each test were recorded every 1000 cycles. For the test with the greatest vibration frequency (40 Hz) this corresponds to a sampling frequency of 0.04 Hz. For the test with the lowest vibration frequency (10 Hz) this corresponds to a sampling frequency of 0.01 Hz. Since the main focus here is the long-term variation of the power, and not its short-term fluctuation, the recorded time-series were smoothed to remove the short-term fluctuations and better expose the underlying long-term trends. Operatively, this was achieved using MATLAB built-in functions (the function ‘smoothdata’, implemented with default settings). A representative example of a power time-series smoothing is provided in figure 4.

To better compare data from the different harvesting devices, the recorded power signals were normalised with respect to the maximum power value measured during the corresponding test:

\[ P_{\text{norm},i}(t) = \frac{P(t)}{\max(P(t))} \]  

(1)

This definition is used to compare the trends rather than the amplitudes of the harvested power. This is deemed useful as the expected power amplitudes will vary significantly between the different devices due to their significantly different mechanical and electrical properties discussed in section 2.1.
3. Results

3.1. Power degradation

PVEHs, as noted previously, are electromechanical systems; so that any degradation in their performance can be attributed to a degradation of their mechanical properties, a degradation of their electrical properties, or a combination of both. In all our tests, natural frequency and optimum load resistance were measured before and after each run, so that their overall variation during the tests can be assessed. The change in natural frequency can be regarded as indicative of the change in mechanical properties of the harvester. The optimum load resistance, on the other hand, depends on the mechanical (frequency and damping) and electrical (capacitance) properties of the harvester, so that any variation can capture both mechanical and electrical degradation effects. Even though it is clearly not possible to separate the effects, monitoring the variations of natural frequency and optimum load resistance can provide useful indications on the variation of the underlying mechanical and/or electrical properties of the harvesters. In what follows, therefore, the recorded changes in natural frequency and optimum load resistance are provided together with the power time-series to help interpret the observed trends.

Figure 5 shows the change in natural frequency and optimum load resistance measured at the end of each test as a percentage of the initial values measured at the start of the test.

As can be seen in figure 5, the PVDF harvesters do not show any detectable variation in natural frequency, whereas a decrease in natural frequency on the order of 3%–9% is observed with the MFC-10, MFC-20, QP-20 and QP-30 harvesters. On the other hand, all harvesters except the MFC-10 experience a variation in optimum load resistance, which increases in all cases except for the harvester PVDF-30.

Figure 6 shows the output power harvested during the tests, whilst summarizing power figures are provided in table 4 and figure 7. As can be noted, the harvested power levels from PVDF cases is the lowest (on the order of 0.1 mW), the harvested power levels from MFC cases is about an order of magnitude higher (on the order of 1 mW), and the harvested power levels from QP cases is another order of magnitude higher (on the order of 10 mW). This is expected due to the differences in mechanical and electrical characteristics of the harvesters discussed in section 2.1.

For the PVDF harvesters, the variation in power output is evident although rather limited in magnitude (within ±19%). Since for the PVDF harvesters there is no significant variation in natural frequency, the observed variation of power output can be traced back to the variation in optimum load resistance. The harvester PVDF-20 experiences the lowest variation in load resistance (+6%), and correspondingly presents the most stable power output during the test. The harvesters PVDF-10 and PVDF-30 experience larger variations in load resistance (+17% and −20%, respectively), and their power output profiles correspondingly show more pronounced variations. The power trends are opposite: decreasing for PVDF-10 and increasing for PVDF-30, and this may be the consequence of the opposite variation of the corresponding load resistance, which increases for PVDF-10 and decreases for PVDF-30. For the PVDF harvesters, therefore, the observed variation in power output seems to reflect the variation in the optimum load resistance.
For the MFC harvesters, the most pronounced variation in power output is observed for MFC-20. For this harvester, there is a variation in both the natural frequency (−5%) and optimum load resistance (+5%). For the harvester MFC-10 there is a variation in natural frequency (−4%) but no detectable variation in optimum load resistance, and the corresponding power output variation is quite limited. Similarly, for the harvester MFC-30 there is a variation in optimum load resistance (+7%) but no detectable variation in natural frequency, and the power output variation is again quite limited. For the MFC harvesters, therefore, the observed variation in power output seems to reflect the variations in the natural frequency and in the optimum load resistance: a larger cumulative variation in natural frequency and optimum load resistance yields a larger variation in power output.

For the QP-10 harvester there is a variation in optimum load resistance (+10%) but no detectable variation in natural frequency, for the QP-20 harvester there is a variation in both the natural frequency (−3%) and optimum load resistance (+34%), and for the QP-30 harvester there is a more pronounced variation in both the natural frequency (−9%) and optimum load resistance (+37%). The variation in power output of the QP
harvesters is milder for QP-10, more pronounced for QP-20 and even more so for QP-30, indicating that a larger cumulative variation in natural frequency and optimum load resistance yields a larger variation in power output.

In conclusion, therefore, for all harvesters the variation in power output can be traced back to the variations in natural frequency and optimum load resistance. As can be noted in table 4 and figure 7, the power output in most tested cases decreases with time, consistently indicating a degradation in performance as consequence of the degradation in mechanical and/or piezoelectric properties. Importantly, by comparing the average power values it is evident that increasing the tip mass does not necessarily increases the average power output. This is particularly clear for the QP harvesters, and suggests that a larger tip mass might exacerbate the degradation of the mechanical and/or piezoelectric properties of the harvester, therefore reducing the power gain that would in principle be expected from having a bigger tip mass.

As noted previously, all harvesters have been tested for three million cycles. After the testing was concluded, each harvester was visually inspected. Despite the recorded variations in power output, visual inspection did not reveal any noticeable sign of mechanical degradation or fatigue. This indicates that the modifications that lead to the power degradation are likely localized in the microstructure of the active elements, and are therefore not observable at the macroscopic scale. This seems consistent with existing studies [51, 53, 54] that identify the formation of micro-cracks in active piezoelectric elements as a potential leading cause of power degradation.

3.2. Power density

Given that the sizes of the employed devices have significant differences, power generation as an absolute measure does not represent a fair comparison metric. As such, power density is defined as the output power after the testing period divided by either the total volume of the harvesting device or by only the piezoelectric layer volume (also see table 1). Another important metric introduced here is the power density to material cost ratio, a metric that could be used to assess power generation cost of the three material options presented in this study. The power density to material cost ratio was obtained by dividing the power density by the cost of a single unit. Note that, the prices used to compare the devices were acquired directly from the manufacturers based on 2019 pricing, and were taken from the cost of a single harvesting device; any materials used in making the device other than the active piezoelectric layer, such as the steel shim and tip masses, were not accounted for in estimating the cost. Using both power density and power density to cost ratio is believed to be helpful in deciding which devices to use for a given need. Here, the comparisons provided consider degradation effects, thus providing a more realistic comparison of devices’ performance. The results are provided in table 5 and figure 8.

As can be seen from table 5 and figure 8, whilst the highest power density is achieved with the QP harvesters, the highest power density to cost ratio is achieved with the PVDF harvesters, indicating that these latter are cost-effective despite the lower power output. Within our tests, degradation effects were clearly seen for all QP cases. QP tests showed that as tip mass ratio increased the power density and power density to cost ratio decreased due
to increasing degradation effects. Nevertheless, QP showed the best power density for given tip mass ratios, figure 8. MFC cases showed greater power density values based on the total volume definition when compared to the PVDF cases even for the MFC-20 case which showed significant degradation. Nevertheless, PVDF cases showed much better power density values when evaluated based on the volume of the piezoelectric layer only.

Figure 8. Comparison of the (a) power density, and (b) power density to cost ratio for tests conducted. (c) and (d) are the same plots as (a) and (b) but on a log-scale for better visualisation of the orders of magnitude.

Table 5. Comparison of power density and power density to cost ratio.

| Test  | Power density (mW cm$^{-3}$) | Power density to cost ratio (mW cm$^{-3}$$^{-1}$) |
|-------|-----------------------------|-----------------------------------------------|
|       | Based on total harvester volume | Based on piezo layer volume | Based on total harvester volume | Based on piezo layer volume |
| PVDF-10 | 0.12 | 1.7 | 0.011 | 0.16 |
| PVDF-20 | 0.21 | 3.0 | 0.021 | 0.30 |
| PVDF-30 | 0.22 | 3.1 | 0.021 | 0.31 |
| MFC-10  | 0.73 | 1.4 | 0.0069 | 0.014 |
| MFC-20  | 0.76 | 1.6 | 0.0072 | 0.015 |
| MFC-30  | 4.5  | 9.0 | 0.043  | 0.086 |
| QP-10   | 14   | 20  | 0.12   | 0.17 |
| QP-20   | 12   | 17  | 0.098  | 0.14 |
| QP-30   | 7.0  | 10  | 0.058  | 0.084 |
Neither the MFC nor the PVDF harvesters were able to show a clear superiority in power density when based on the active layer volume, with the PVDF-10, and PVDF-20 cases showing better values whereas the MFC-30 case showing higher power density compared to its corresponding PVDF case. MFC cases showed the least values of power density to cost ratio, indicating they are the least favourable choice when cost is considered.

3.3. Active tuning of natural frequency
Since the power degradation can be traced back to the variations of natural frequency and optimum load resistance, an obvious compensation strategy would be to actively tune the natural frequency and load resistance during operation. A preliminary assessment of the feasibility and potential of active frequency tuning during operation is presented below. Figure 9 shows the excitation frequency and power output of harvester QP-30, which, as previously discussed, was tested under single-frequency constant excitation (note: this is the same result shown in figure 5). Also included in figure 9 are the measurements obtained for QP-30 when tested whilst actively tuning the excitation frequency to match the changing natural frequency. In this latter test, both the power output and the natural frequency were measured periodically. When a shift of at least 1 Hz was measured for the natural frequency, the input frequency to the shaker was manually adjusted to compensate for the measured shift (manual adjustments are highlighted with a red marker in figure 9).

As can be noted, at first the active frequency tuning is beneficial in terms of harvested power. This is no longer the case towards the end of the test, however, and the harvester actually broke at about 200 minutes. Even though the results in figure 9 are no more than a preliminary proof of concept, they seem to indicate that active frequency tuning may be beneficial for energy harvesting in the short term, whereas in the long term it may exacerbate the mechanical degradation of the harvester thereby shortening its mechanical life. Clearly, active frequency (and electrical load) tuning should be properly investigated, but this goes beyond the scope of this study.

4. Concluding remarks
We analysed and compared the power degradation performance over prolonged operation of PVEHs realized using PVDF, MFC, and QP: three of the most frequently used piezoelectric materials in energy harvesting applications. Overall, we realised and tested nine PVEHs: three different piezoelectric options (PVDF, MFC, and QP) and three different tip mass ratios (tip mass ratios of 10, 20, and 30). The harvesters, unimorphs of cantilever beam geometry configuration, were tested under single-frequency excitation for three million vibration cycles, observing power degradation that ranged from a few percent to 60%. During testing, the excitation amplitude was kept constant at 1 g, whilst tip masses were added to the harvester so that the vibration frequencies varied within 10–40 Hz: a range representative of low-frequency vibration applications. The observed variations in
harvested power reflect the variations in natural frequency and optimum load resistance of the harvester, thereby linking the degradation in performance to the degradation in mechanical and/or electrical properties of the harvester. Tip masses have a direct effect on the mechanical and/or piezoelectric properties of the harvester, therefore affecting the power gain that would in principle be expected from having a bigger tip mass. Whilst the highest power density is achieved with the QP harvesters, the highest power density to cost ratio is achieved with the PVDF harvesters, indicating that these latter are cost-effective despite the lower power output. Future research should concentrate on potential strategies to mitigate power degradation of the harvesters, such as the active tuning of the natural frequency and/or electrical load during operation.

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