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Biodegradable materials in acoustic energy harvesting with triboelectric nanogenerator

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Abstract

The growing global demand for energy requires sustainable and renewable energy alternatives to fossil fuels. This study investigates acoustic energy harvesting using a triboelectric nanogenerator (TENG) fabricated from biodegradable materials. TENG has shown encouraging results in harvesting mechanical energy to electricity even with low-powered energies. Acoustic energy is considered as low-powered energy and has low energy density, making its conversion into usable electricity challenging. Nevertheless, acoustic energy remains ubiquitous and presents potential to act as renewable energy source.

TENG operates through triboelectrification and electrostatic induction. Phenomena is based on contact and separation, where two materials with different electron affinities generate charge. In this work, biodegradable polylactic acid (PLA) and cellulose acetate (CA) films of varying thicknesses were examined through this phenomenon. Thicknesses were characterized using a texture analyzer with an oscilloscope to evaluate the influence on energy output. The optimal pair produced 7.7 V under texture analyzer test conditions. Using the same material thicknesses, acoustic energy harvesting yielded a maximum of 1 V at 123 Hz. The level of voltage output is almost sufficient for sustaining microchips and tiny electronics such as sensors.

The results indicate that further optimization of the setup and material surface modifications could significantly improve the total energy output of TENG. This study highlights the potential of novel biodegradable material pair working in acoustic energy harvesting. Also, acoustic energy harvesting is evaluated in developing self-sufficient energy solutions for low-power electronics and sensor systems.

Keywords Triboelectric nanogenerator, acoustic energy harvesting, low-powered energy, biodegradable materials, self-sufficient energy, sustainable development

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Tiivistelmä

Energian tarve kasvaa jatkuvasti kehittyvässä yhteiskunnassa, jossa digitalisaatio ja liikenteen sähköistyminen lisäävät tarvetta entisestään. Samaan aikaan tulee huomioida ympäristöystävälliset tavat tuottaa energiaa, jotta fossiilisten polttoaineiden käyttö saataisiin minimoitua maailmanlaajuisesti. Tämä kandidaatintyö käsittelee äänienergian talteenottoa tribosähköisellä nanogeneraattorilla (*triboelectric nanogenerator*, TENG), joka on valmistettu biohajoavista materiaaleista. Äänienergia tunnetaan matalatehoisena ja usein hukkaan menevänä energiamuotona. Se on lähes kaikkialla läsnäolevaa, jonka vuoksi se voisi toimia yhtenä ympäristöystävällisenä energialähteenä. Äänienergian suurin ongelma kuitenkin on sen alhainen energiatiheys, joka tekee sen talteen otosta haastavaa.

Tämä kandidaatintyö tutkii TENGin kykyä talteenottaa matalatehoista äänienergiaa. TENGlle ominaista on sen kyky generoida mekaanisesta energiasta sähköenergiaa. Esimerkiksi meren aallot, ihmisten liike tai tässä tapauksessa ääniaaltojen mekaaninen värähtely voidaan muuntaa sähköenergiaksi. Käytännössä TENG toimii tribosähköisen efektin ansiosta, jossa kaksi eri elektroniaffiniteetin omaavat materiaalit kohdatessaan polaroituvat vastakkaismerkkisesti. Nämä sähköiset potentiaalit muuntuvat induktion myötä sähkövirraksi, joka voidaan hyödyntää energialähteenä mm. pienelektroniikassa, sensoreissa tai erilaisissa lääketieteellisissä sovelluksissa.

Koska äänienergia on hyvin matalatehoista, TENGin toiminta tulee optimoida mahdollisimman tehokkaaksi esimerkiksi modifioimalla materiaalien pintoja. Monissa tutkimuksissa on huomattu, että modifikaatiot parantavat kontaktipinta-alaa ja tehostavat elektronien siirtoa pinnalta toiselle, jotka parantavat TENGin tehokkuutta. Lisäksi TENGin materiaalien paksuudet tulee määrittää ihanteellisiksi, jotta jännitealuetta saadaan kasvatettua. Materiaalien paksuus määrittää niiden ominaisvärähtelytaajuudet sekä sen, kuinka tehokkaasti elektronit voivat siirtyä materiaalin pinnalta toiselle.

Tämä kandidaatintyö koostuu kirjallisuuskatsauksesta sekä kokeellisesta osuudesta. Työ yhdistää TENGin perusominaisuudet, sen erilaiset modifikaatiot sekä äänienergian talteenoton, joita tarkastellaan kirjallisuuskatsauksessa. Työn kokeellisen osuuden tarkoitus on arvioida ja osoittaa uuden biomateriaaliparin kykyä toimia hyvänä materiaaliparina äänienergian taltioinnissa. Työssä esitellään kahden biohajoavan materiaalin, polymaitohapon (*polylactic acid*, PLA) ja selluloosa-asetaatin (*cellulose acetate*, CA), kalvojen eri paksuuksia.

Materiaalien paksuudet karakterisoitiin tekstuurianalysaattorilla, jossa hyödynnettiin oskilloskooppia. Materiaaliparin parhain tulos oli 7.7 V tietyillä paksuuksilla. Tämä materiaalipari valittiin äänienergian maksimaalista talteenottoa varten, jonka tulos nykyisellä testiasettelulla oli parhaimmillaan 1 V 123 Hz taajuudella. Jännite riittää ylläpitämään pienjännitteistä elektroniikkaa sekä mikropiirejä, mutta ei takaa tehokasta energian varastointia. Varastoinnin hyötysuhde jäisi hyvin alhaiseksi ja vain muutamia mikrovoltteja saataisiin talteen. Tuloksia tarkasteltaessa kuitenkin huomattiin, että optimoimalla mittausjärjestelmää voidaan saavuttaa korkeampia tuloksia, joten biomateriaaliparilla on potentiaalia toimia TENGin komponentteina.

Äänienergian talteenoton tutkimusalue on aktiivisesti kehittyvä ja vain muutamia kymmeniä julkaisuja on tehty aiheesta. Tutkimusalue on tärkeä, koska se tukee kestävän kehityksen periaatteita ja voisi mahdollistaa esimerkiksi omavaraisen energiantuotannon pienelektroniikassa sekä energian varastoinnin akkuihin.

Avainsanat Triboelektrinen nanogeneraattori, äänienergian talteenotto, biohajoavat materiaalit, omavarainen energia, kestävä kehitys

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Abbreviations

TENG	Triboelectric nanogenerator
PLA	Polylactic acid
CA	Cellulose acetate
PTFE	Polytetrafluoroethylene
PVDF	Polyvinylidene fluoride

1 Introduction

The demand of sustainable and renewable energy sources has continuously grown in the last few decades. At the same time, fossil fuels usage is wanted to go down and more devices are fabricated with biodegradable materials. Consequently, new innovations and biodegradable materials are needed to cover a wider range of energy harvesting. For example, sound energy harvesting has been investigated to power small scale electronic devices and self-powered systems which could help the energy harvesting to go towards sustainable applications. [1] However, even crashing waves, concert halls and traffic noise sound waves have extremely low-pressure fluctuations which are hard to furthermore harvest as energy.

Triboelectric nanogenerators (TENGs) have promising properties to convert acoustic energy to electricity. [2] When two materials with different surface affinities get in contact, electrons transfer from one surface to another. This causes opposite charges and potential differences between materials, which generate electric current when materials are separated. These are also called contact-separation TENGs, and they have features to harvest low-powered and renewable energies. As a result, devices are considered as green energy harvesters while they are also low-cost, lightweight and can be fabricated from a wide range of materials. TENGs are also widely applied from nanoscale to large scale applications, particularly for self-powering nanosensors, wearable devices and other internet of things (IoT) systems. [3]

The main challenge in TENGs lies in achieving proper contact-separation between materials, especially with low-powered energies. Because of this, many material properties must be balanced to enhance the energy output from acoustic energy harvesting. If the properties are poorly characterized, it may lead to low energy output and the devices' inefficient working. TENGs' materials biodegradability and durability are also considered while enhancing the properties of acoustic energy harvesting. [3]

This thesis presents two biodegradable materials and their capability to work as acoustic energy harvester materials in TENG. In literature review, TENGs' function in acoustic energy harvesting is explained and its drawbacks are discussed more. After that, materials are presented and characterized in the experimental part. Lastly, TENGs future for acoustic energy harvesting is discussed and evaluated by these materials.

2 Triboelectric nanogenerator in acoustic energy harvesting

Triboelectric effect has long lasting roots in physics and material chemistry. It is one of the earliest observations in material's electric properties and tribology, and yet its phenomena and working mechanism debate are still going. [4] In 2012 Wang et al. [5] research group developed triboelectric generator which has been further examined by other research groups ever since. Generator has gained the name triboelectric nanogenerator after it has shown promising development under nanoscale effects and structures in sensing and harvesting fields.

This section presents first triboelectric nanogenerator's (TENG's) contact-separation working mode and properties to take account while fabricating TENG. Focus is kept in thickness and dielectric properties. After that, few biodegradable materials are presented and evaluated to work in TENG. Finally, acoustic energy harvesting is analysed from harvesting point of view.

2.1 Structure and mechanism of triboelectric nanogenerator

The main principle of TENG is based on triboelectrification and electrostatic induction. Triboelectrification is the base for materials to charge opposite polarities against each other through contact. One material which charges positively is called tribopositive material. Other material which charges negatively is called tribonegative material. Mechanical energy is therefore converted to electricity through electrostatic induction. In this state, mechanically charged materials affect surrounding electric fields that force electrons to move in cables and creates an electric current. Through this, alternating current occurs when contact and separation happens continuously. [6] This process is demonstrated in Figure 1.

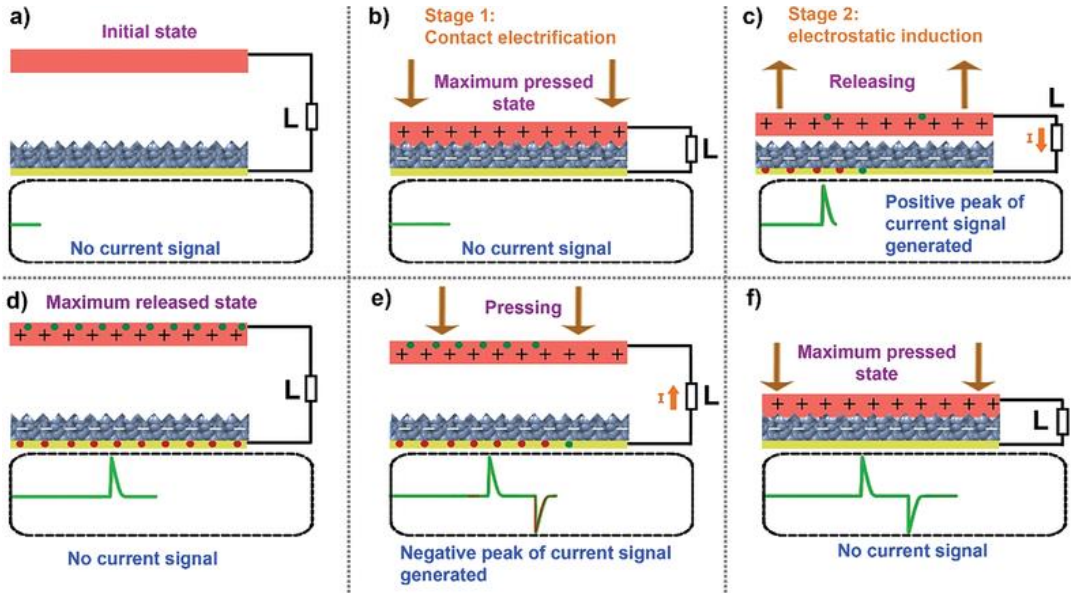

Figure 1. TENG's contact-separation mode generates alternating current. [7]

As previously mentioned, many parameters affect the efficient working of TENG. One of the key aspects is electron affinity. [6] It is crucial to choose materials with different electron affinities so the triboelectrification can occur. Triboelectrification happens based on which material can donate electrons and which accepts them. Basically, a wider electron affinity gap between the materials enhances the efficiency of electron transfer from one surface to another.

Because the TENG basic mechanism is based on triboelectrification, contact area has a critical role in TENG's final energy output. [1] High contact area enables more electrons to travel from surface to another per contact-separation cycle. This leads for higher charge transfer and therefore higher energy output.

At the same time material dimensional properties play a crucial role, such as thickness and size. [8] A deeper explanation to these is in 4.4 section. Nevertheless, TENG's dynamic behavior is also one important parameter when considering efficiently working TENG. In TENG working, dynamic behavior means for instance frequency and its impact for the energy output. If contact-separation happens in higher frequency, it enables higher charge transferring between materials. [9] As figure 1 shows, alternating current occurs when materials are pressed and separated, therefore higher frequency in contact-separation leads for more frequent alternating current.

In summary, TENG's working principle is dependent on many parameters for example contact area and electron affinity. When more parameters from above are taken account, the more efficient TENG become and higher voltage can occur. Before continuing to explore acoustic energy harvesting, it is crucial to understand which materials would be most suitable in TENG. In other words, next chapter discusses about choosing the biodegradable pair combination for TENG.

2.2 Biodegradable materials in triboelectric series

Materials' triboelectric features are usually presented in triboelectric series. As already mentioned, triboelectric effect can only occur between two materials that have different electron affinities. For this type of categorization, materials are usually sorted in triboelectric series that give a basic idea of which two materials would be the best against each other when brought in contact. [10, 11] There are still other parameters to take account such as: flexibility, mechanical properties and thermal properties, because all the material properties affect electron transfer from surface to another and proper contact area.

Usually, other than biodegradable materials are used in TENG. [12] Figure 2 shows the most common materials in TENG where electron acceptors and donors are shown separately. For instance, most commonly used tribonegative material is teflon, polytetrafluoroethylene (PTFE) and most commonly used tribopositive material is aluminium (Al).

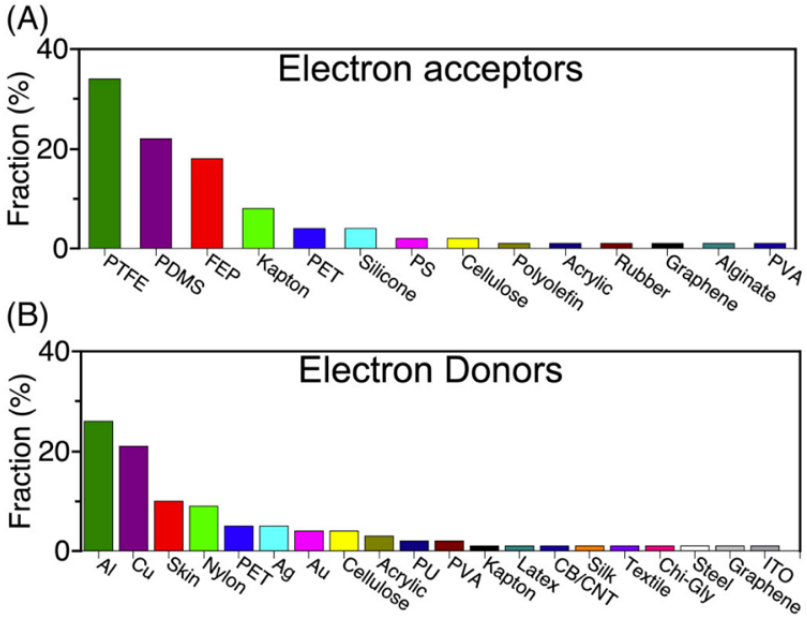

Figure 2. Common material choices for TENG as acceptors and donors in 2020. [12]

As Figure 2 shows, most of the time biodegradable materials are not used in TENG. This can be because most of the biodegradable materials have stability problems and are usually moisture sensitive. These properties reduce the energy output and can cause charge loss. Also, biodegradable materials may not have that long service of life, and they lose the ability work as efficiently as in the beginning. [13] Nevertheless, many biodegradable materials are dielectric materials and have great potential to cover non-biomaterials in TENG.

For instance, polymers are widely used in TENGs due to their excellent dielectric properties, which allow them to store and maintain surface charges efficiently. [12] The main reason for charge storing is their ability to polarize in response to electric field. This means that material's electron cloud shifts when material is brought to electric field. Shifting happens mostly in strongly polarizable groups such as halogens, aromatic π-electrons and strong permanent dipoles, such as nitro, carbonyl, and hydroxyl groups. In other words, the more polymer contains polarizable groups, the more efficient charge storing occurs in material after contact-separation. This directly enhances the triboelectric output by increasing the surface charge density. [14]

Dielectric materials work as capacitors. In other words, the charge storing in material can be related to capacitors. [8] While thinking the polarizability of the functional groups in capacitors, at the same time material's thickness matters. Capacitor equation (1) is presented below.

$$C = \varepsilon_r \varepsilon_0 \frac{A}{d} , \quad (1)$$

where ε_r is the relative permittivity, ε_0 permittivity of vacuum, A surface area of the material and d thickness of the dielectric material. Relative permittivity is also presented as dielectric constant for materials. Basically, what this thesis evaluates in materials and methods sections are the relative permittivity and thickness. If the relative permittivity increases, the higher is the charge storage in material and higher voltage can occur through contact-separation. At the same time, if the thickness reduces, material has higher charge storaging.

Polylactic acid (PLA) is one of the most widely used and produced bioplastic [15]. PLA is known for its renewable forming process. It is usually made from plant starch and has favorable film formation properties. PLA can be proper choice for TENG, because it has also great mechanical and dielectric properties. For instance, its dielectric constant ε_r, relative permittivity, is ranging 2.5–3.0 and the structure contains polarizable groups. Few articles show PLA as one component in TENG, for example in Pan *et al.* [16] research. TENG reached up to 500 V at best against gelatin. In addition, PLA is usually classified as tribopositive material, but it stands in the middle in the triboelectric series.

Another great option as biodegradable material in TENG would be cellulose acetate (CA). Also Figure 2 shows that, not only the CA is used in TENG but also other cellulose derivatives are in great development in TENG fabrication [17]. CA is derived from cellulose where hydroxyl groups are partly or fully replaced with acetate groups which increases the structures polarizability. Also, CA has outstanding properties in film formation, mechanical properties and dielectric features which make it even more favorable choice in TENG. CA's dielectric constant is 3–5 depending on how acetylated the CA is. It is relatively higher compared, for example with PTFE that has only 2.1 dielectric constant.

In addition to PLA and CA, various natural and eco-friendly alternatives have been investigated [13], including plants (and their components), hair, sugar, cellulose, spider silk, and egg white. Joo et al. [18] demonstrated that the tribopositive properties of TENGs can be enhanced to improve energy output. Their study specifically examined keratin, a protein obtained from human hair and animal fur, as a coating material. These materials should be more investigated while they are environmentally friendly and show promising results in enhancing TENG's energy output. Bio-based and biodegradable materials offer numerous advantages in TENG's function making them potential to replace other non-biomaterials [13].

Next chapter discusses more about the acoustic energy harvesting and harvesting by itself. Few of the main drawbacks of sound harvesting are evaluated and few solutions to them are presented. In addition, many of the previous parameters are thought when fabricating sound-driven TENG.

2.3 Acoustic energy harvesting

TENG has shown promising development through every mechanical energy conversion [19]. This has provided also sound harvesting and sensing field improvement. Sound harvesting and sensing includes auditory sensing, voice recognition, ultrasonic and low-frequency sound harvesting. These categories are shown in Figure 3.

Figure 3. Different applications of TENG in sound harvesting and sensing till 2024. [20]

Figure 3 presents that TENG can be used widely in different applications for sound sensing and harvesting. It is also improved in many areas of industries such as wearable devices, health monitoring, internet of things and even in smart home devices. [19]

No matter which application in sound sensing and harvesting is in question, the major challenge in sound lies in low energy density of acoustic waves. [1] For instance, typical ambient sound levels, such as normal conversation, range between 60–70 dB, corresponding to only about 0.0001–0.001 W/m² of energy. For comparison, sun energy power is reaching 1000 W/m² on bright day, so the sound wave's energy is million times weaker.

Furthermore, sound waves disperse as they travel through open space, which reduces their intensity. [21] According to the inverse square law, when the distance from the source is doubled, the sound intensity decreases to one-quarter of its original value. In addition, sound waves travel in every direction, which means again lower intensity in one direction. [21] Some improvements on the material surfaces are needed to make sure that even lower intensity sound waves could be caught.

In addition, usual ambient sound across a wide range of frequencies at the same time. [1] According to literature, natural resonance frequencies in any device vary with several parameters such as thickness, damping, geometry and size. Film thickness plays a crucial role in finding the optimal frequency range that guarantees the best operation of TENG.

For instance, Zhou et al. [2] review about ranging frequencies in sound harvesting suggests that more information about these parameters are needed to understand more about the natural frequencies of the materials. One possible solution would be to choose thinner films for sound harvesting, because they tend to vibrate more readily also in higher frequencies. [2] At the same time, vibrating films do not achieve as proper contact area as direct mechanical force. Contact occurs only at oscillating points of the films which lead to less effective contact area.

To improve these drawbacks of sound waves, Fan et al. [22] introduces 0.125 mm thin paper-based TENG. Maximum power density reaches up to 121 mW/m² under 117 dB and it has the ability to charge a capacitor at the rate of 0.144 V/s from human speaking. Basically, researchers have taken into account many parameters such as thickness, geometry, size and enhanced contact area, when developing the TENG. In addition, they have chosen Cu and PTFE by their difference in electron affinities. Figure 4 presents the ultrathin rollable paper-based TENG.

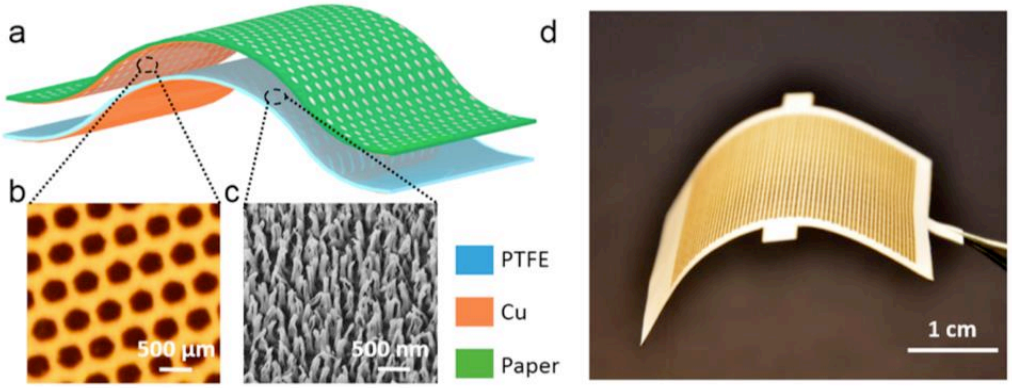

Figure 4. a) Ultrathin rollable sound harvester. b–c) Surface patterning on both materials. d) 0.125 mm thin paper-based TENG.

As previously discussed, enhancing contact-area between materials improves TENG efficiency and increases the energy output. Figure 4 b–c) show great example of how to enhance contact area. Other material has holes and other pikes that can go through the holes. When materials get in contact the spikes go through the holes and electron transferring can happen more efficiently from surface to another. This is also a great example of how micro and nanostructures effect on contact area and can enhance the energy output in TENG.

Another example of enhancing TENG properties for sound harvesting is to double the basic working mechanism in vibrating membrane. For instance, Cui et al. [23] proved that sandwich structured TENG with metal mesh top layers reached 90 V open-circuit voltage and could light up 138 LEDs. TENG also works stable in broad range from 50 Hz to 425 Hz with 100 dB sound source. Sandwich structure is presented in figure 5.

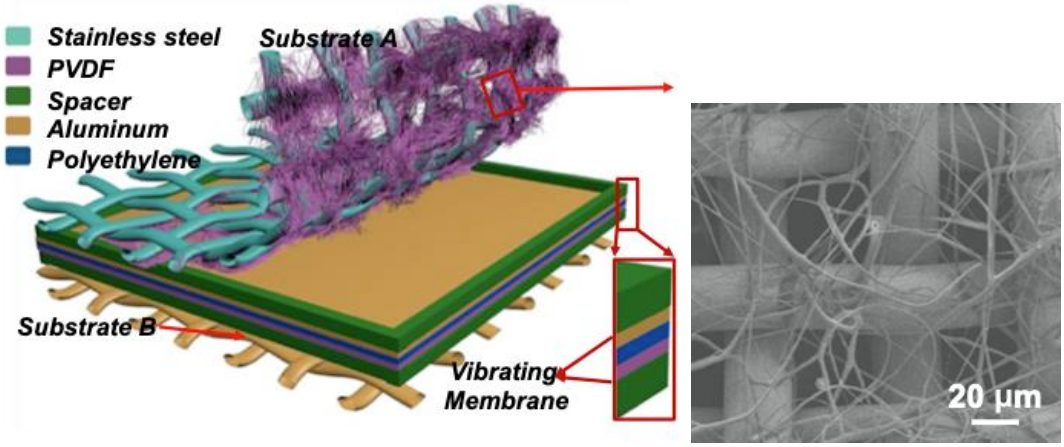

Figure 5. Fabricated TENG and SEM image of PVDF nanofibers on stainless steel mesh. [23]

Substrate A and substrate B have mesh structure with PVDF nanofibres, modified surface, that allows better air circulations in the device than normal flat surface. This improves the pressure fluctuations on vibrating membrane between the substrates and increases the contact area. This also helps even lower intensity sound waves to travel and vibrate the surface. A vibrating membrane between substrates is presented in Figure 6.

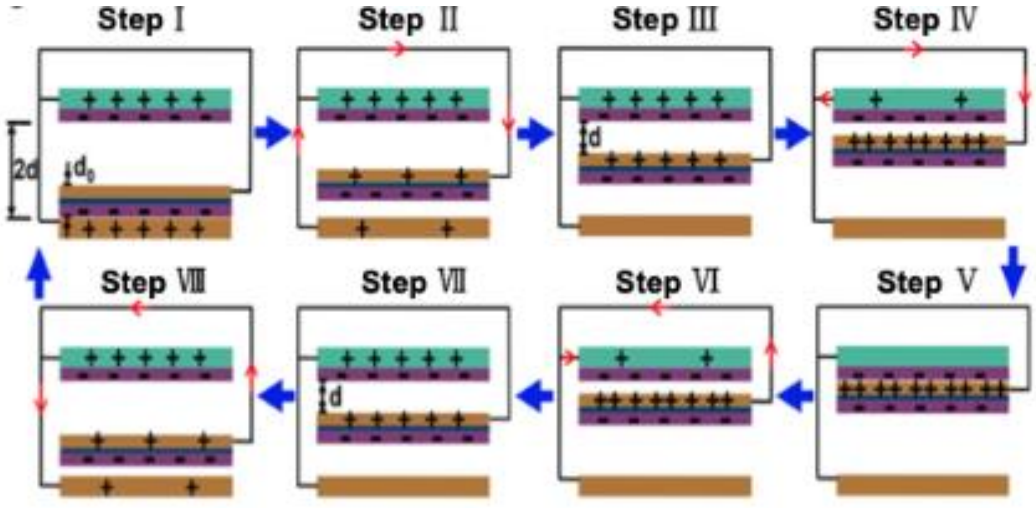

Figure 6. Mechanism of sandwich sound driven TENG. [23]

Sandwich TENG has two simultaneous contact-separation and overcomes the basic contact-separation TENG. Basically, this enhances the chance for more efficient energy harvesting compared to the basic contact-separation effect. As can be seen, vibrating membrane get charged both positively and negatively, because both tribopositive and tribonegative materials are separated by inert polymer. This provides better charge transfer from vibrating membrane to substrates [23].

In summary, these two articles were both very differently fabricated TENGs for sound harvesting, and yet, both gave decent results as harvesters and sensors. Both have taken into account that sound waves are hard to catch and have low energy density. Other has modified thickness, contact area and shape while other has exploit 3D nanostructures and has multilayered the contact-separation mode. In other words, both has taken into account different properties and still got great results based on the application. Still, the lack of biodegradable materials remains common in both approaches.

Next section of materials and methods presents same type of evaluations of parameters. For example, material's shape, size and thickness are characterized to their optimized version for enhancing the energy output. Also, the material's dielectric properties and electron affinities were thought while starting to design the TENG from biodegradable materials.

3 Materials and methods

Novel triboelectric material pair is introduced in this section. PLA and CA were chosen as the tribo-pair because they combine biodegradability with excellent dielectric characteristics. In addition, this pair has never been reported in any literature as a pair.

This section contains film fabrication and materials characterization. Because experimental part consisted of two phases: texture analysing and sound harvesting, both results are presented below in row. First, film fabrication is introduced for texture analysing and then for sound harvesting. After that characterization setups are shown in same order.

3.1 Fabrication of films

Solutions of both, PLA and CA, were 5 % w/v in their solvents. Solutions were casted into 9 cm diameter glass petri dishes and dried on leveled surface. After the films were fully dried, they were pealed with 0.1 M sodium hydroxide and washed with water. PLA and CA films are shown in Figure 7.

Figure 7. PLA as a round film on the left side and CA cut as square film on the right side.

As Figure 7 presents, PLA and CA are both similar looking. Both are transparent and have little wavy structures. Nevertheless, both have great film forming properties and are easy to bend. Films were then cut to 4 by 4 cm square. For making films conductive on other side, copper tape was placed to the films as smoothly as possible to prevent wrinkles. Copper tape was also used as a conductive tail for the samples. Fabricated samples can be seen in Figure 8.

Figure 8. CA and PLA films cut to 4 by 4 cm samples and covered with copper tape on the other side.

Some samples were modified physically and chemically. For example, plasma etching, rubbing the surface with sandpapers to have smoother surface and chemical binding with PVA were used. The aim for these modifications was to enhance the energy output and have efficient contact area between materials, because macro/nano structures usually enhance the contact area between materials and may help in electron transferring [24].

Before testing the samples with a texture analyzer, masking tape was placed on top of the copper tape. It prevented the wrinkling of the films while testing them multiple times. These samples were used to characterize surface modifications and film thicknesses. Furthermore, best samples were fabricated again for the sound harvesting.

Film fabrication for sound harvesting was made only for the best two samples, PLA and CA, that gave the best results with texture analyzer. PLA and CA samples were sputtered with Au/Pd coating to make them conductive on other side. This remained the flexibility of the films. Samples were cut to 7 cm diameter and then glued to spacers. When samples are in contact, diameter is 6 cm. The samples were fabricated round, because sound waves can travel more evenly through the sample. The samples are shown in Figure 9.

Figure 9. CA and PLA films attached to 0,2 mm spacers and sputtered with Au/Pd coating to make the surface conductive.

3.2 Characterization

4 by 4 cm fabricated films were set against each other for contact-separation testing using a texture analyzer. First, the parameters were set to cause 50 N force from 2 mm distance for 1 cycle in pre-test speed 1 mm/s, test speed 1 mm/s and post-test speed 1 mm/s. After that, distance near 40 N was selected from 50 N chart and parameters were changed to pre-test speed 40 mm/s, test speed 40 mm/s and post-test speed 40 mm/s for 500 cycles. All the data was saved through oscilloscope.

The probe used for the measurements was P/25P. Glass Petri dishes were set on the probe and on the surface to get smooth contact-separation for the films. Films were then stuck to the Petri dishes with double-sided tape. This setup can be seen in Figure 10.

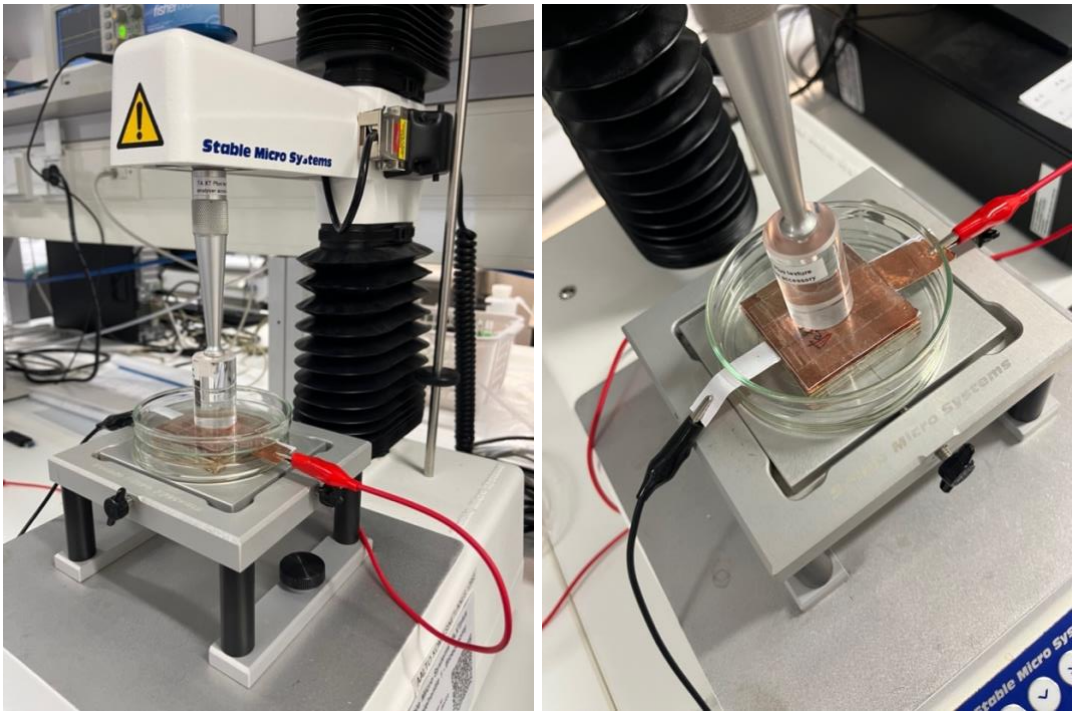

Figure 10. Contact-separation setup.

The setup was attached to an oscilloscope through a bread-board with 1 MΩ load. The voltage output depended on the type and size of materials. Later in the result section, voltage output is expressed as voltage range. Voltage range is read from voltage peaks from highest to lowest.

This setup was utilized to characterize sample thicknesses and different surface structures. After finding the best pair with the best thicknesses, samples were benchmarked with different forces, distances and frequencies. In addition, different sizes were tested to see how much size affect the voltage output.

For sound harvesting the same kind of contact-separation setup was built from one 4 cm height plastic cylinder and cork ring. The cylinder was placed under the samples near to the speaker. On top of that, samples were placed in sequence: CA, spacer, PLA. Then samples were 4 cm away from the speaker. Cork ring was placed on top of the samples and the space was kept open. This setup can be seen in Figure 11.

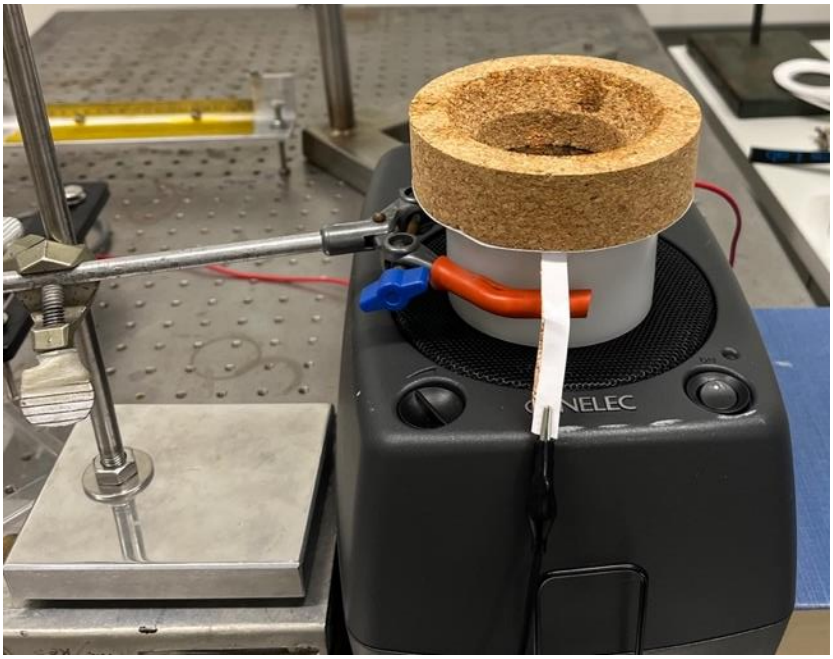

Figure 11. PLA against CA sample in sound harvesting setup.

Both samples were tested against PTFE to track the best natural frequencies. Samples were set to vibrate through 50 Hz – 350 Hz frequency range and best frequencies for PLA were 84 Hz, 95 Hz, 97 Hz, 98 Hz and 104 Hz, and for CA 106 Hz, 110 Hz, 119 Hz and 123 Hz.

Also, three different distances were tested to see how much the sound intensity would decrease. In all the experiment, the same volume was used, and they were set to 50 % on computer and 50 % in software. This setup was one of the earliest testing setups and the upper cork ring needed to be held manually stable. This way the best voltage outputs were obtained.

4 Results

This section presents best results of new tribo-pair's thicknesses' characterization and sound harvesting. In 4.1 section materials thicknesses are characterized for sound harvesting. In 4.2 section a novel tribo-pair of PLA and CA is presented and in 4.3 section best sound harvesting results are presented. At the end in 4.4 section, results are evaluated and discussed through attention to parameters that effects on voltage output.

4.1 Film thicknesses

CA thicknesses were measured from 0.01 mm – 0.1 mm and PLA thicknesses were measured from 0.03 mm – 0.13 mm with caliper. Different thicknesses were set against reference materials Al and PTFE to see which thicknesses are the best and then further on selected to sound harvesting. Thicknesses were characterized with texture analyzer (Figure 10). In Figure 12, CA and PLA thicknesses are presented.

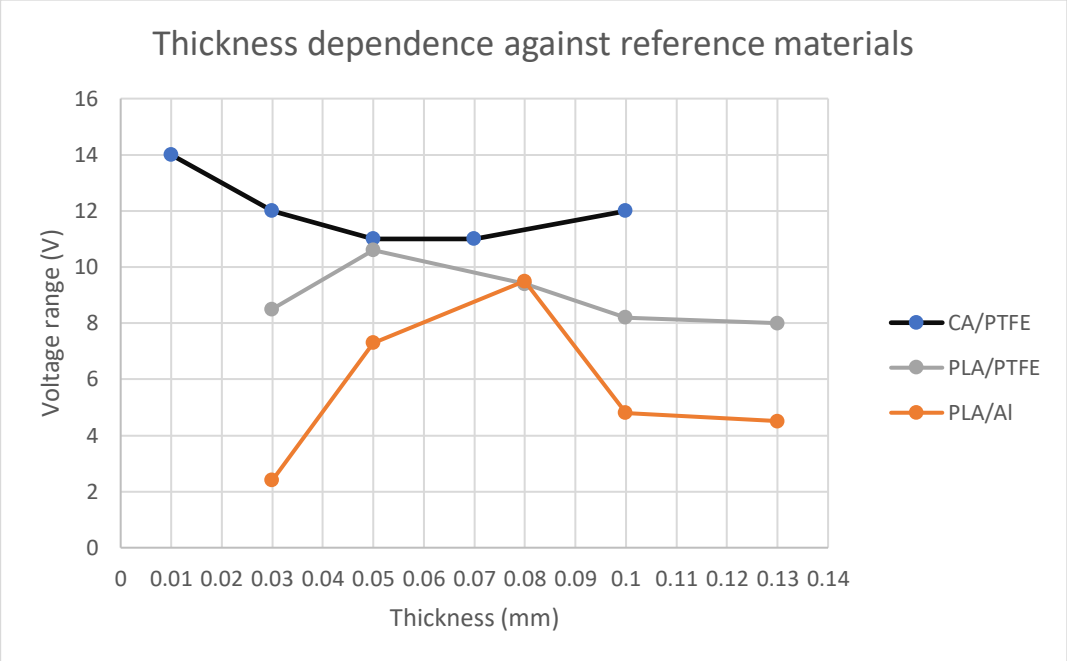

Figure 12. CA and PLA thicknesses against reference materials. Black curve is CA thickness against PTFE. Grey curve is PLA thickness against PTFE. Orange curve is PLA thickness against Al.

Black curve shows that when CA thickness increases, voltage range decreases. The highest voltage output of 14 V is reached when thickness is 0.01 mm. Also, CA could reach higher in voltage outputs compared to PLA. This could mean that CA is more tribopositive material against PTFE than PLA.

Grey curve shows that when PLA reaches 0.05 mm thickness, voltage range starts to decrease. The best thickness for PLA against PTFE is 0.05 mm with 10.6 V voltage range. Also, the same pattern can be seen in orange curve. Orange curve shows that when PLA reaches 0.08 mm, voltage range starts to decrease. The best thickness for PLA against Al is 0.08 mm with 9.6 V voltage range.

In addition, 0.08 mm thickness is spot where grey curve and orange curve cross. In other words, PLA gives same voltage output against tribopositive and tribonegative reference materials. This states that PLA could work as tribopositive and tribonegative material based on the opposite material pair.

The best thicknesses for CA and PLA were obtained. For CA, the best thickness was 0.01 mm and for PLA it ranges between 0.05–0.08 mm. These thicknesses improved the energy outputs and were chosen to work in sound harvesting setup.

4.2 Novel tribo-pair

PLA and CA were set against each other in texture analyzer setup (Figure 10). Data was collected after 500 cycles of conditioning, so the pair had more time to gain charge on films. As previously discussed, continuous contact-separation generates alternating current. This can be read as voltage range from highest peaks to lowest peaks from oscilloscope. Voltage range is presented from best PLA/CA pair in Figure 13.

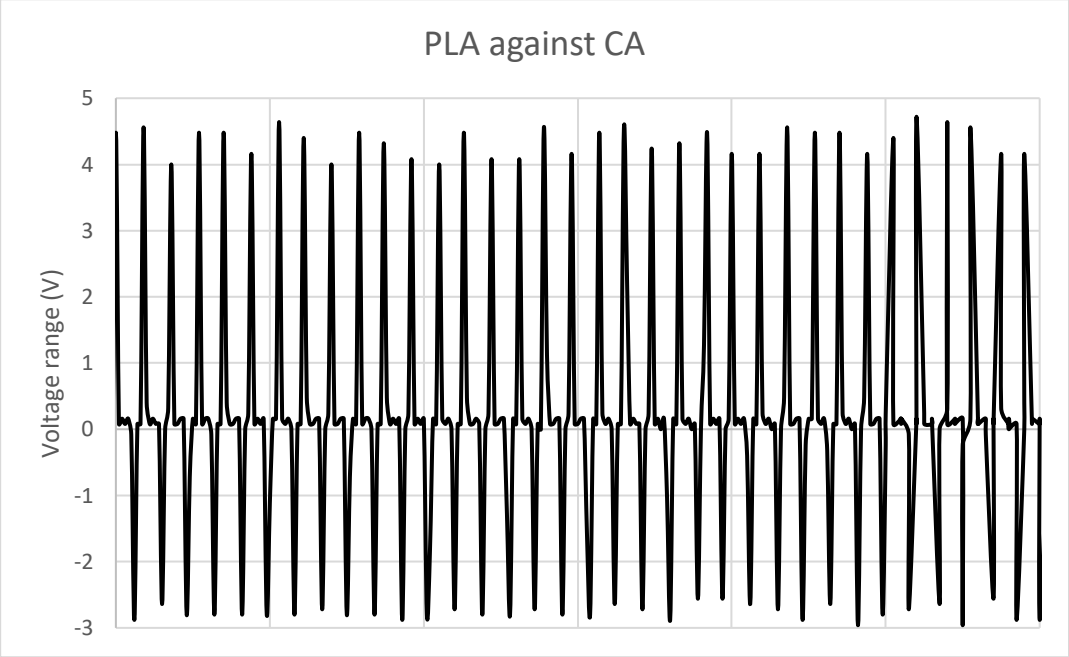

Figure 13. PLA against CA voltage range in the end of 500 cycles.

The setup generated 7.7 V across the 1 MΩ load. PLA thickness in this pair was 0.08 mm and CA thickness was 0.02 mm. Generally, best results in texture analysing were obtained (in Figure 12) when PLA thickness varied between the best thicknesses 0.05–0.08 mm and CA thickness 0.01-0.02 mm.

This suggest that repeatable samples vary by their micro/nanoscale surface structures and still can generate same range of voltage outputs. At the same time, PLA and CA pair works differently than reference materials. They show different surface structures and properties which lead variations in thicknesses and voltage outputs. In addition, it is essential to understand how much PLA and CA pair can generate voltage with direct mechanical force. It gives an idea what voltage output is achievable before continuing to sound harvesting experiments.

4.3 Sound harvesting

As in characterization section explained, different natural frequencies were tested for both PLA and CA with sound harvesting setup (Figure 11). The best outputs for PLA against PTFE were in 95 Hz and for CA against PTFE in 123 Hz. These frequencies were most stable and had repeatable results that reached almost 1.5 V. Based on these results, PLA and CA were set against each other, and the following results were obtained at the same frequencies 95 Hz and 123 Hz. Results are shown in Figures 14 and 15.

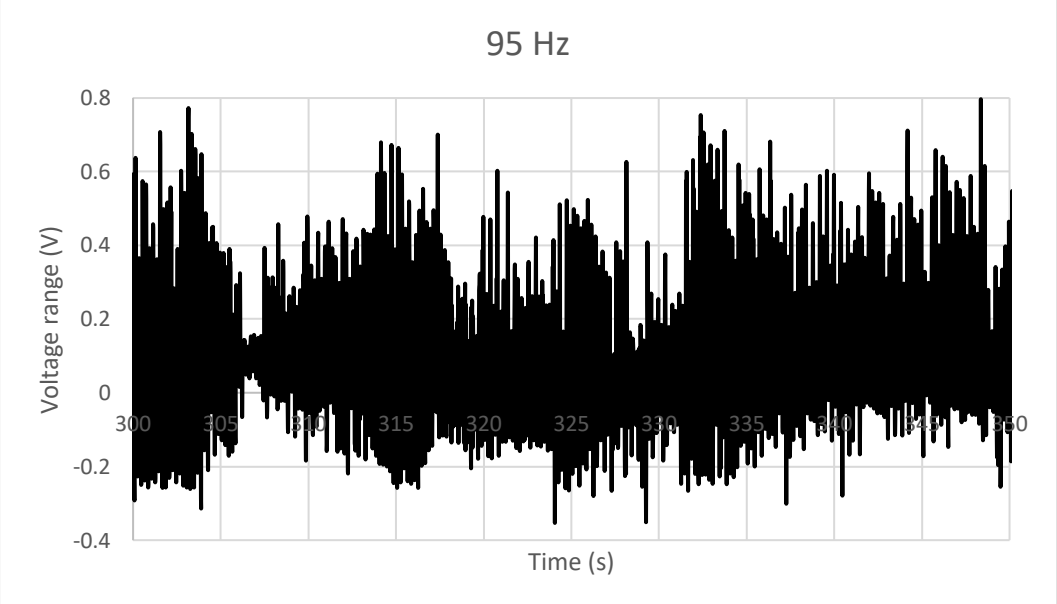

Figure 14. PLA against CA in 95 Hz for 350 seconds.

Because the upper cork ring needed to be held manually stable, sound harvesting results were unstable at some points. Unstable voltage range can also be because the films vibrate and then the contact area is sometimes less and sometimes higher. However, Figure 14 shows that highest peaks are reaching to 0.8 V, but most stable range is approximately in 0.5 V. Lowest peaks reach almost to -0.4 V, but most stable range is in -0.2 V. When adding the most stable parts together, voltage range with 95 Hz is 0.7 V.

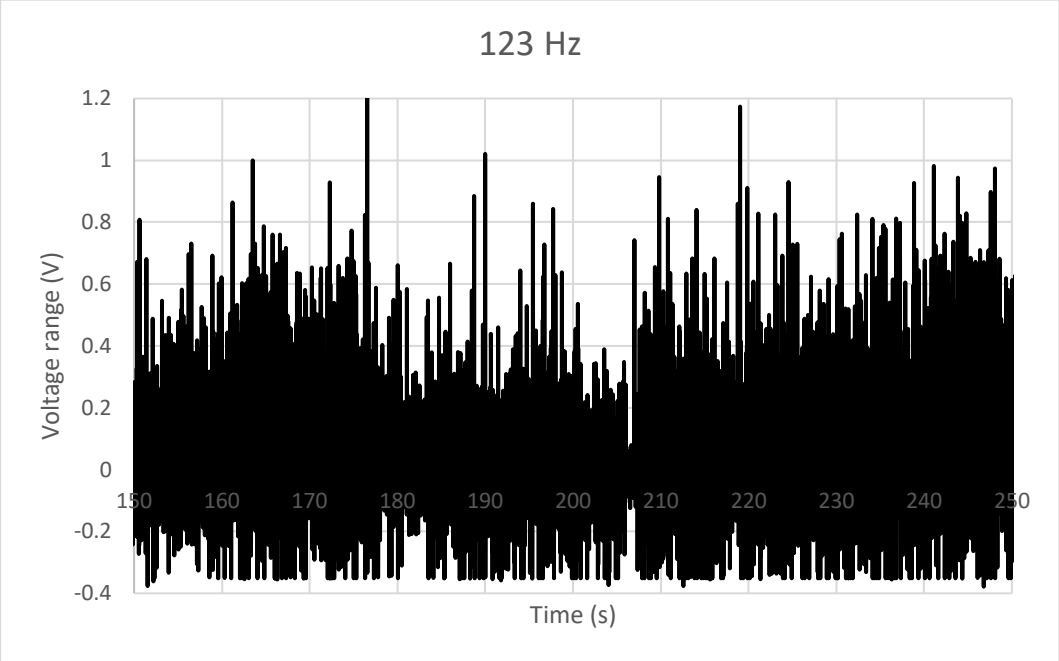

Figure 15. PLA against CA in 123 Hz for 350 seconds.

A similar pattern as 95 Hz of unstable voltage range is observed in 123 Hz. It is more stable than frequency 95 Hz. This occurs because the CA sample was thinner sample, and this was also CA's natural frequency. Basically, this would mean that CA vibrates more readily [2] against PLA so higher and more efficient contact area is generating more stable voltage.

However, highest peaks are 1.2 V, but most stable range is in 0.7 V. Lowest peaks reach almost to -0.4 V, but most stable range is in -0.3 V. When adding the most stable parts together, voltage range with 123 Hz is 1 V. The setup generated 1 V across the 1 MΩ load. Relatively low voltage output occurs because films have lower contact area based on the low energy density sound waves and vibrating films. Also unoptimized test setup was used and humidity conditions were 60–70 % when the sound experiments were done.

4.4 Discussion

Meanwhile preparing films, humidity conditions were changing between 35–70 %. Best film formation happened in 35–40 % and problems in the texture analyzing results started to appear after 50 % humidity was crossed. For instance, Nguyen et al. [25] research shows that humidity is one crucial feature that reduces the voltage output in TENG. This occurs, because humidity leads to surface discharge and thereby reduces the voltage range. Basically, water works as a thin conductive layer and therefore charges cannot accumulate high in films. In addition, humidity effect on film's robustness and mechanical properties. [25]

Based on humidity conditions of the date, new results for the same films were caught. Result variation happened between 2 V – 7.7 V in texture analysing. For instance, CA and PLA pair reached even higher results in voltage than CA and PTFE pair on same date. This would mean that PLA would be more tribonegative material than PTFE, which is not the case based on their electron affinities.

For PLA thickness, two main results were obtained. Firstly, PLA gave the same results against positive and negative reference materials in 0.08 mm thickness. This means that PLA can work as tribonegative and tribopositive material which is a great finding from biodegradable material point of view. PLA has potential to work against other material pair combinations in TENG.

Secondly, energy output increased until PLA thickness reached a certain point. The same pattern for PLA thickness is observed in literature [16]. These results are not in straight line with equation 1 (in chapter 2.2), which states that when thickness increases, charge storage decreases. Now it is the opposite till a certain point. In other words, PLA loses the charge in lower thicknesses and when the thickness is optimized charge storage in material is maximized. Beyond this point, higher thickness starts to disturb the electrons movement and decreases the triboelectrification on the surfaces.

For CA the same pattern for thickness can be seen in the literature. When thickness reduces the higher the voltage gets. Usually, when materials thickness reduces, electron transfer between materials gets easier. There is a shorter distance for electrons to travel between material surfaces and less repulsion. With shorter distance, electrons can reach the other surface faster and they can travel more freely. [8]

As previously mentioned, CA has acetyl groups that have high polarizability. This enables better charge storaging and can be the main reason why CA behaves differently than PLA. Another possible explanation for this can be the dielectric material's trap states [26]. Trap states are located in the bandgap. Trap states capture the electrons and holes and thereby restricts their movement between the valence and conduction bands. This reduces the charge transfer and higher charge can generate in material.

Basically, this would mean that CA have deeper trap states than PLA which can bind charge more efficiently. In other words, PLA has narrower trap states where electrons can easier escape. This would explain why CA can perform more efficiently even in thin layers, whereas PLA often requires an optimal thickness to achieve its best triboelectric performance. However, more quantum mechanical research needs to be done to understand the electron flow and charge storaging in these films.

As earlier mentioned in film fabrication (3.1 section), some surface modifications were done to samples, because surface modifications are known to enhance triboelectric effect [27, 28]. These did not give significant improvements on voltage output, so they were not presented. In other words, if plain CA gave 6.5 V against PTFE, with surface modification it remained the same or reduced the voltage.

Drawback for the sound distance was reduced by placing the plastic cylinder under the samples and near to the speaker. This way the cylinder could confine the soundwaves straight to the samples and sound intensity loss was reduced. Anyhow, results were not fully reaching their full potential, because the setup was not optimized. As previously explained (in section 4.3), the upper cork ring in the setup needed to be held manually stable to get better vibration on the films. Lower contact area is the main reason for low voltage range and when the films vibrate, they do not maintain full contact while vibrating against each other.

To combine all the results from basic mechanism of TENG, biodegradable materials, contact area, sound harvesting and energy outputs, more research needs to be done in this specific pair. Because TENG development started in 2012 [5], it is crucial to examine all parameters through different material pairs. Table 1 presents comparison for different kind of TENGs and their applications. It compares materials pairs, contact area and voltage outputs. All the TENGs are fabricated to work in contact-separation mode.

Table 1. From different materials fabricated TENGs, their contact areas, applications and voltage outputs.

Material 1	Material 2	Contact area (cm^2)	Application	Voltage output (V)	
PLA	PTFE	4 x 4	Contact-separation	9.5	-
CA	PTFE	4 x 4	Contact-separation	14	-
PLA	CA	4 x 4	Contact-separation/Sound harvesting	7.7/1	-
PVDF	Nylon	5 x 5	Sound harvesting	546	[29]
Cellulose nanofibrils	Fluorinated ethylene propylene	1 x 1	Contact-separation	5	[30]
Egg white	Chitin	1 x 2	Medical science	8	[31]
Dry leaf (powder)	PVDF	4 x 4	Wind energy harvesting	560	[32]

Table 1 gives an idea of how much the voltage outputs varies based on the TENG fabrication, which materials are used and in which application. In blue background, PVDF and Nylon show great potential to work as sound harvesting materials. Results reached for 546 V in resonant frequency [29]. It even reaches near to other renewable energy source, wind power in red background, which has way more power density in mechanical force for contact-separation.

However, wind energy harvesting TENG's one component is biobased and yet the results reach surprisingly high. Feng et al. [32] used dry leaf and PVDF material properties and succeeded to find great material pair for TENG. Materials have compatible chemical and surface structures for enhancing the contact area, great dielectric properties to generate higher voltage and different electron affinities for efficient electron transfer.

In green background, there are fully bio-based TENGs in different applications. As can be seen, the energy output is low even if they are generated with normal mechanical force and not with low energy sound. Also, the contact area is approximately 16 times smaller. Material pair and size matters not only for sound and resonant frequencies, but also in triboelectrification. The reason for this lies in biobased materials electron affinities. They usually are located in the middle of the triboelectric series, so the electron affinities are near each other. Then electron transferring cannot happen that efficiently.

In yellow background, these is this thesis's results. When comparing the results to others, they are low to consider the size and application. This means that the full potential of these materials is not used. Future work can contain improvements in contact area and surface modifications, because the pair has promising material properties such as biodegradability, dielectric properties and differing electron affinities. They showed great improvement only in thickness optimization, so by estimation, they could reach even higher, when other parameters are optimized.

5 Conclusions

This thesis examined the potential of PLA and CA to work as harvesters in sound-driven TENG. Highest stable voltage reached 1 V in 123 Hz with characterized thicknesses. Testing setup was not fully optimized, and many parameters affected the voltage output. These properties were for instance contact area, material size and humidity. With fully optimized material parameters and setup, TENG would be more efficient.

Biomaterials show great potential to work as sound harvesting materials. Many of them have great dielectric properties that provides better usage for harvesting any energy with TENGs. While biodegradable materials offer environmental benefits, challenges occur such as material stability, short service life, and lower power density compared to non-biodegradable materials [13]. Again, many parameters need balancing for enhancing the energy output of biobased TENGs. However, many biodegradable material combinations have already shown great results [17, 31], and they would ensure more environmentally friendly TENG and sustainable energy yield.

PLA as one biomaterial, showed great potential to be an active compound in TENG. It can work as tribonegative and tribopositive material, and its properties should be further examined. Since PLA showed different behavior in thickness compared to CA, its dielectric properties should also be studied in more detail, for example using quantum mechanical approaches. This could provide more information about dielectric features of PLA and furthermore easier way to modify for TENG.

Further examination could be done with these films to see if they really could work as harvesters. For example, how efficiently PLA and CA pair could harvest the sound and store in capacitors. Although the films produced promising voltage even with unoptimized setup, further experiments are necessary. If proper charging results could be harvested, then it would be essential to continue to degradation and film durability testings. Finally, the practical energy harvesting must be efficiently done.

Even though, sound energy has low energy density, and it disperses in open areas, there is already great observation about enhancing the sound-driven TENGs properties with multilayered structures or 3D structures on surfaces [22, 23, 29]. Also, the most effective way to guarantee the highest energy output with sound is to install the TENG near the sound source. For instance, industrial machines or loudspeakers provide favorable conditions for sound harvesting TENG, because they produce high volume sounds and usually vibrates in same frequencies. TENG layout could provide simplified TENG preparation and allow more concentrated parameter improvements.

In summary, TENG's development is a new research field in energy harvesting, and it is growing through the global demand of new energy sources. PLA and CA material pair show strong potential to generate sufficient voltage output with sound-driven TENG, even with unoptimized setup and as biodegradable materials. At minimum, material pair could work as a sound wave sensor, but for acoustic harvesting more optimized and characterized setup is needed. With further optimization, this system would give higher and more stable results. In addition, PLA and CA material pair should be more investigated, because they have never been reported as TENG pair in literature.

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