Effect of Microorganism on Flax and Linen

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Abstract
In this paper, an attempt has been made to study the biodegradability of flax and linen when the said materials undergoes soil burial test. In this technique, the microorganisms are responsible for the biodegradation of fibres. The degradation of material being analyzed through naked eye to electron microscope, through IR-spectroscopy and thermal analysis. The time period taken by the microorganism to consume completely the natural fibre is also studied.

Keywords: Flax; Linen; Microorganism; SEM; FTIR; TGA

Introduction
Degradation is a process of breaking down a material into its constituent elements by a physical, chemical or a biochemical process which should be irreversible. When this process of degradation is aided by the attack of living matter especially microorganisms, resulting into mineralization or biomass, it becomes Biodegradation [1]. Biodegradation of a material is takes place in three different stages i.e., biodeterioration, biofragmentation and assimilation.

Bio deterioration of materials is a combined result of lots of degradative factors like mechanical degradation, thermal degradation and degradation due to the presence of moisture, oxygen, ultra violet light and environmental pollutants. Due to the result of these mentioned factors, a huge amount of microorganisms stick onto the surface of materials. Biofragmentation is a process in which microorganisms increase their population and secrete enzymes and free radicals, which break down macromolecules to oligomers and monomers. In assimilation, energy, new biomass and various metabolites used by microorganisms are produced and simple gaseous molecules and mineral salts are released into the environment [2].

There are two types of biodegradation, aerobic and anaerobic. When material is biodegraded in the presence of oxygen it is called aerobic biodegradation and if without oxygen then anaerobic biodegradation. For a material to be completely biodegraded it must be converted into mineral salts and carbon dioxide, water and minerals and the intermediate products should contain biomass [1]. Both the aerobic and anaerobic types of biodegradation are represented in below mechanisms:

I. \( \text{C}_\text{polymer} + \text{O}_2 \rightarrow \text{CO}_2 + \text{H}_2\text{O} + \text{C}_\text{biomass} + \text{Salts} \)

II. \( \text{C}_\text{polymer} \rightarrow \text{CO}_2 + \text{CH}_4 + \text{H}_2\text{O} + \text{C}_\text{residue} + \text{Cbio} + \text{Salts} \)

Where \( \text{C}_\text{polymer} \) represents either a polymer or a fragment that is considered to be composed only of carbon, hydrogen and oxygen. When \( \text{C}_\text{polymer} \) is completely converted into gaseous products and salts, then the biodegradation is completed [1].

The biodegradation of materials depend upon the polymer chemistry and the environment in which they are exposed. Some of the important factors that directly influence the rate of biodegradation are presence of microorganism, availability of oxygen, availability of water, temperature and pH etc. [1]. Microorganisms attack on any kind of fibre or material surface in various steps like 1) Microorganisms stick onto the surface of a material either by adhesion or aggregation, 2) proliferation of attached microbial cells, 3) production of enzymes, 4) biodegradation of materials, 5) reduction of degree of polymerization of material polymer and production of degradable products [2].

The degradation rate of cellulose and cellulosic textile substrates mostly depends on microorganisms used. Bacteria and fungi are the two main groups of microorganisms responsible for enzymatic degradation of cellulose. In the presence of bacteria the degradation of the cellulose fabrics proceeds from the surface towards the inside, In the presence of fungi, after the revival of the cuticle, the organisms penetrate through the secondary wall into a lumen where they grow [3]. The main function of the enzymes is to decrease the degree of polymerization, resulting in damaging the structure of the fibres and the fibres loses their strength. The rate of degradation of cellulose is directly related to its degree of crystallinity. Hence for amorphous cellulose having less degree of crystallinity is more susceptible for enzymatic degradation than a crystalline one. The degradation rate also depends on other parameters like degree of orientation, degree of substitution and presence of non-cellulosic substances [3]. So in this present study, an attempt has been made to understand the process of biodegradation of flax and linen fibres when they are subjected to soil burial test.

Materials
The materials were used for the present study of biodegradation are flax and linen fabric which are shown in Figure 1.

Experimental Procedure
In this experiment samples of all types of fabrics were cut into

![Figure 1: Photo image of Flax and Linen fabric.](image-url)
pieces of 5 × 5 cm² and four samples were taken from each kind of fabric and they were buried in soil according to the ISO 11721-1:2001 and ISO 11721:2003 standards. The samples are buried in such a way that all four pieces of one type of fabric are buried in one separate beaker of 1000ml, so that different materials may not mix with each other. After every week soil from all beakers is taken out and moisturized with distilled water, after that soil with samples is again put back in the beakers and one piece of fabric from every kind of textile material is kept out to study the effect of microorganisms. These samples are first rinsed in ethanol/water (70%/30% volume fraction) solution for approximately 10 min before drying at room temperature and after that further experiment were conducted.

**Evaluation by NIKON ECLIPSE (E200) optical microscope**

NIKON ECLIPSE (E200) is equipped with high-resolution camera and image analysis software. The measurements were performed according to a pre-defined macro, which ensured that all samples were analyzed in the same way and under the same conditions. It has several optical lenses marked as 10X, 20X and 40X having magnification range from 100 to 400 times.

**Evaluation by scanning electron microscope (SEM)**

The samples were examined under Scanning Electron Microscopy [SEM] using Zeiss EVO 18 Special edition for imaging analysis. Examination was done with working distance (WD) of 6.5 mm and Electron Gun frequency at 20 kV for imaging at a magnification of 4KX with SEM Workstation.

**Evaluation by fourier transformation infrared spectroscopy (FTIR)**

Infrared (IR) spectroscopy is one of the most important and most frequently used analytical technique which enable interpretation of the chemical structure of the substance consequently identification of its functional groups. A sample is placed in the path of infrared beam and the functional groups of sample absorb different infrared frequencies and this absorption causes vibrations of the molecules.

**Evaluation by thermogravimetric analysis (TGA)**

In Thermogravimetric analysis the apparatus type that is used in EXSTAR6000 (TG/DTA 6300) instrument at a heating rate of 10°C/min with a range from 25°C to 500°C. The sample that is to be run on this machine is heated at constant rate, while change in mass of sample is recorded as function of temperature. The weighing of the sample is done by a thermo-balance which is present inside the furnace.

**Results and Discussions**

**Microscopic analysis of Flax and Linen**

The sample of flax and linen fabric that was used to study is woven, bleached and mercerized. It was cut into four pieces so that after every week one piece can be taken out to study the degradation effects by using various mentioned techniques. Then flax and linen samples before experimentation and taken out from soil after seven, fourteen and twenty one days have been analyzed visually and with the help of optical and scanning electron microscope. The findings are pictorially represented in Figures 2 and 3 as follows.

If we look at the samples with naked eye there seems minor change in the samples. This is because of the two reasons, first the mass per unit area of the fabric is too high and secondly the fabric is blended with polyester fibres which shows very little or no effect of degradation. But if we observe microscopic visuals then we come to know that the major portion of cellulose have been degraded by the microorganisms.

The biodegradation of linen fabric was quite quick and after two weeks it was extremely difficult to separate linen fabric from the soil. Photographic representation of linen fabric after for two weeks. Samples of Linen fabric are the one who were severely attacked by microorganisms and only after two weeks it was difficult to separate the fabric samples from the soil. The so quick degradation effects from the soil burial test are because of the structure of the linen fabric, as the fibers are not tightly twisted in the yarns.

**FTIR analysis of Flax and Linen**

The FTIR investigation of three week behavior of flax and linen samples when taken out every week and studied by FTIR is represented by the Figure 4 as follows.

The FTIR spectra of flax fibers shows intensive absorption in the region 1600-1720 cm⁻¹ which is caused by stretching vibrations of carbonyl groups. It is known that the exact position of these groups depends on their conjugation with benzene rings (in this case the position is lower than 1700 cm⁻¹). In the case without conjugation the position is higher than 1700 cm⁻¹. The other reason for the appearance of these bands can be the presence of some impurities in the initial fibers, such as fats, waxes, and resins. Two intensive bands with...
maxima at 2850 and 2918 cm$^{-1}$ are attributed to deformation vibrations of C-H groups in methyl and methylene groups [CH$_3$, CH$_2$, CH$_2$-OH] belonging to cellulose as well as to lignin. The shape of this band is not typical of cellulose, which usually exhibits three-shoulder band with a maximum at 2900 cm$^{-1}$ in this region. Moreover, the band with a maximum at 2900 cm$^{-1}$ exhibits typical cellulose shapes [4].

The prominent change which shows that the material has degraded can be seen by the spectra in the range of 1400 cm$^{-1}$ to 1650 cm$^{-1}$, here an increase in the intensities of the spectrum after every week has been noticed. It represents that the initial functional groups that were present in the linen sample has been attacked by microorganisms, degraded and they have converted them into new biomass. At 1637 cm$^{-1}$ the peak of 2nd week is most prominent which is representing the absorption of water molecules; it shows that with the passage of time structure of cellulose collapses and absorption of water molecules increases [5]. According to reference [6] the spectra of cellulose show decrease of bands particularly at 1372 cm$^{-1}$, 1336 cm$^{-1}$, 1313 cm$^{-1}$, 1280 cm$^{-1}$, 1160 cm$^{-1}$ and 1105 cm$^{-1}$ when moving from high crystalline to amorphous cellulose, which means that the samples are degraded [7].

**TGA analysis of flax and linen**

In Thermogravimetric analysis for flax and linen the maximum temperature is set to 600°C and 500°C respectively and the ramp rate is set to 10°C per minute. The weight of the sample taken should be very small, in the range of 5mg to 10mg. The reduction in weight percentage versus increase in temperature plot for flax and linen samples are shown in Figure 5. It has very interesting curves (in the case of flax) at temperatures in the range of 350°C and 450°C. The first bending in the curves at round about 350°C shows the conversion of cellulose into carbon dioxide [8-10], ash and complete evaporation of water. The second dip in the curves that ends at round about 450ºC shows the start of melting of polyester fibres [11-13]. It is clear from the graph that the cellulose portion of the fabric has been degraded.

Again in the case of linen from the Figure 5 that when the linen sample before degradation was tested by TGA, maximum amount of fabric was burnt and the remains was only the ash. But in case of linen sample after two weeks due to the exposure to soil, microorganisms have degraded the samples and the amount of cellulose has been reduced [14,15]. So when the sample is heated up to 500°C, it reduced...
the weight up to 50% but after that it was impossible to reduce the mass as there was not cellulose left. The above graph shows that the weight loss percentage for fabric taken out of soil after two weeks is much less as compared to the fabric that has no contact with the soil. These are the clear signs that sample has been biodegraded after two weeks.

Concluding Remarks

The biodegradation of flax and linen fabric under the attack of microorganisms present in the soil were studied for several weeks and the changes that occurred in the samples were measured by different scientific methods and approaches. But if we look at the results linen seems to be damaged much quicker than Flax. The degradation happens may be due to the development and attack of microorganism during prolonged contact of fibre with soil.

References