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# Radio-frequency Ar plasma treatment on muga silk fiber: correlation between physicochemical and surface morphology

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## Abstract

Radio-frequency (RF) Ar plasma treatment is carried out on natural muga silk fibers in a capacitively coupled plasma reactor. The physical and thermal properties of the muga fibers are investigated at an RF power of 20 W and in the treatment time range of 5 to 20 min. The virgin and plasma-treated muga fibers are characterized by Fourier transform infrared spectroscopy and X-ray photoelectron spectroscopy. The effect of Ar plasma treatment can be observed only on the outermost layer of the muga fibers without any significant variation in their bulk and thermal properties, as supported by differential scanning calorimetry and thermogravimetric analysis. Improvement in tensile strength and hydrophobicity of the plasma-treated muga fibers is observed at lower treatment time and RF power. Attempts are made to correlate the properties of the plasma-treated muga fibers with their surface chemistry and surface morphologies.

**Keywords:** Radio-frequency plasma, Muga silk fiber, Peptide chain

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## Background

Extensive research is carried out on the radio-frequency (RF) plasma treatment of natural silk fibers by modifying their wetting, dyeing, and printing properties; tensile properties; shrink resistance; flame retardance; anti-bacterial properties; biocompatibility; hydrophilicity; and hydrophobicity [1-7]. RF plasma treatment is a dry and clean process and does not suffer from any environmental and health concerns. It has a major advantage over the conventional wet chemical process in terms of reduction of waste and pollution problems and conservation of chemicals, water, energy, and time. RF plasma treatment on silk fibers with non-polymerizable precursors, such as inert gases, only influences their outermost surfaces by increasing or decreasing the surface roughness up to few-nanometer skin depths. Removing contaminants like pollutants, oxide layer, etc. from the surface and also etching the weakly bonded components

from the fibers can easily be achieved by plasma treatment [4].

Among various natural silk fibers, muga (*Antheraea assamensis*) has the highest traditional and economical importance and is mainly cultivated in the northeastern part of India. Muga silk is generally used for making garments with a variety of conventional designs and embroidery. The muga silk fiber is mainly composed of two sub-fibers, each of which is known as fibroin [8,9]. The two fibroins are surrounded and cemented together by a glue-like protein termed as sericin. As a natural silk, it is mainly composed of a protein which has multiple H bonds in its beta sheet secondary structure. As compared to all other natural silk fibers, muga silk possesses unique properties such as inherent natural golden color, highest tensile strength, high durability, stain free and moisture absorbent qualities, anti-flammable and anti-bacterial properties, biocompatibility, etc. [9-11]. All these properties of muga silk fiber make it one of the most suitable and important candidates for use as a biomaterial with further enhancement and refinement of some of its properties such as tensile strength and hydrophobicity.

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It is a well-known fact that in RF plasma, the properties of fibers are strongly dependent on the external discharge parameters [12,13]. The desired physical and chemical properties of plasma-treated fibers can be obtained through proper control of the external discharge parameters such as input RF power, treatment time, working pressure, gas flow rate, etc. Due to the complexity of the plasma, it is also necessary to study the plasma discharge characteristics (electron temperature, plasma density, plasma potential, etc.) and monitor the active species generated in the plasma, using some useful diagnostic techniques so that one can be able to correlate the plasma parameters with the properties of the fibers. This is essential so far as the industrial and technological applications of plasma-treated fibers are concerned. Besides, through proper *in situ* diagnostics of the discharge characteristics and replicating them by controlling the external plasma parameters, one can successfully reproduce the same preferred qualities of the fibers. In this regard, self-compensated Langmuir and emissive probes and optical emission spectroscopy (OES) technique are often preferred to diagnose the RF plasma discharge [12-20]. On the other hand, OES is a simple non-intrusive optical technique that allows easy identification of different emitted species and deduction of semi-quantitative density trends of emitting species as a function of the experimental variables.

In this work, the RF plasma treatment on muga silk fiber at various treatment times is reported. The objective of this work is to increase the durability of the RF plasma-treated muga silk fiber by enhancing its tensile strength. It is important to mention that to enhance the mechanical strength of muga silk fiber, people generally and traditionally use starch on silk yarn before and after weaving the cloths which is known as 'starching.' This kind of process destroys the quality of the silk product as well as causes many kinds of skin diseases. Inert gas plasma treatment on the silk surface is a promising way to keep its all inherent properties the same as well as to enhance the tensile strength. The improvement in the hydrophobicity of muga fiber in order to reduce the application of natural/artificial dye on the fiber and to make it more hygienic to the skin and comfortable to wear is another motivation of this work. The work also aims at investigating the RF plasma discharge characteristics to better understand the properties of the plasma-treated fibers. The performances of the virgin and plasma-treated muga fibers have been evaluated using various characterization techniques. The correlation of the results obtained from the plasma discharge characteristics and characterization techniques with the observed properties of the fibers is attempted.

## Results and discussion

### Plasma diagnostics

The values of  $T_e$ ,  $n_e$ , and  $n_i$  as obtained from self-compensated Langmuir probe analyses are measured to be 6.7 eV,  $3.9 \times 10^8 \text{ cm}^{-3}$ , and  $4 \times 10^8 \text{ cm}^{-3}$ , respectively. Similarly, plasma potential ( $V_p$ ) measurement from the self-compensated emissive probe is calculated as 16.90 V. During Ar plasma treatment, the DC self-bias voltage that is developed on the RF electrode is measured to be -50 V.

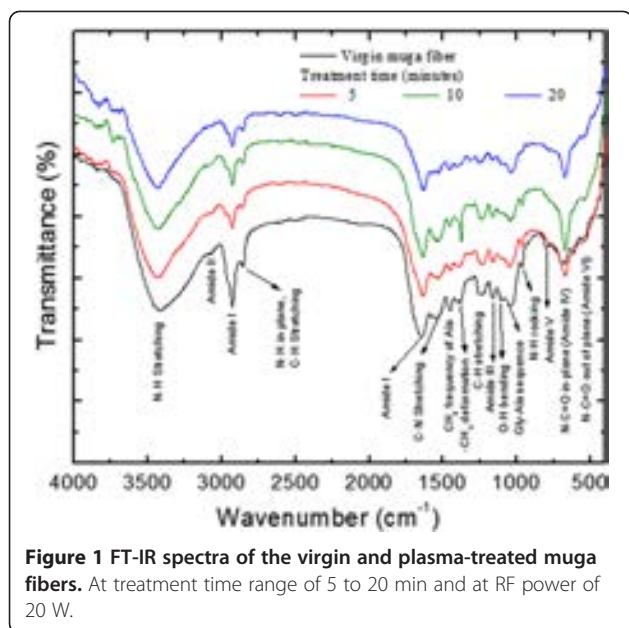
A typical optical emission spectrum is obtained from Ar plasma discharge at an RF power of 20 W. The main transitions that are observed in the spectrum are reported in Table 1. It is revealed from Table 1 that the Ar plasma discharge mainly consists of Ar I species which originates due to dissociation of neutral Ar atoms by electron impact. The detection of only Ar I spectral line indicates that the Ar plasma is free from any contaminant or impurity present in the plasma chamber that may affect the observed properties of the plasma-treated fibers.

### Fourier transform infrared spectroscopy

Figure 1 shows the FT-IR spectra with assigned peaks for the virgin and Ar plasma-treated muga fibers. As seen from Figure 1, the FT-IR spectra show characteristic bands at  $1,645 \text{ cm}^{-1}$  (amide I) and  $800 \text{ cm}^{-1}$  (amide V) which are due to the beta conformation of the crystalline region, while the band appearing at  $1,534 \text{ cm}^{-1}$  (amide II) is due to the random coil conformation of fibroin molecules. The peaks that appeared at  $1,230$  and  $1,380 \text{ cm}^{-1}$  are attributed to the presence of an amino acid group (amide III) and get superimposed by a hydrocarbon functional group, e.g., C-H stretching and  $\text{CH}_3$  deformation of the silk. The appearance of the absorption band at  $684 \text{ cm}^{-1}$  (N-C = O in-plane bending) and  $543 \text{ cm}^{-1}$  (N-C = O out-plane bending) confirms the presence of alpha-form sequential alanine polymer. The peak positioned at  $960 \text{ cm}^{-1}$  signifies the

**Table 1 Transitions of Ar I species during RF Ar plasma discharge**

Species	Wavelength (nm)	Transition
Ar I	383.0	$3s^2 3p^5(^2P_{1/2}^0)4s$ to $3s^2 3p^5(^2P_{3/2}^0)6p$
	425.0	$3s^2 3p^5(^2P_{3/2}^0)4s$ to $3s^2 3p^5(^2P_{3/2}^0)5p$
	706.8	$3s^2 3p^5(^2P_{3/2}^0)4p$ to $3s^2 3p^5(^2P_{3/2}^0)6s$
	751.0	$3s^2 3p^5(^2P_{1/2}^0)4p$ to $3s^2 3p^5(^2P_{1/2}^0)4d$
	767.0	$3s^2 3p^5(^2P_{3/2}^0)4p$ to $3s^2 3p^5(^2P_{3/2}^0)4d$
	772.4	$3s^2 3p^5(^2P_{1/2}^0)4s$ to $3s^2 3p^5(^2P_{1/2}^0)4p$
	794.9	$3s^2 3p^5(^2P_{1/2}^0)4s$ to $3s^2 3p^5(^2P_{1/2}^0)4p$
	801.7	$3s^2 3p^5(^2P_{1/2}^0)4p$ to $3s^2 3p^5(^2P_{3/2}^0)6s$
	811.3	$3s^2 3p^5(^2P_{3/2}^0)4s$ to $3s^2 3p^5(^2P_{3/2}^0)4p$
	826.0	$3s^2 3p^5(^2P_{1/2}^0)4s$ to $3s^2 3p^5(^2P_{1/2}^0)4p$

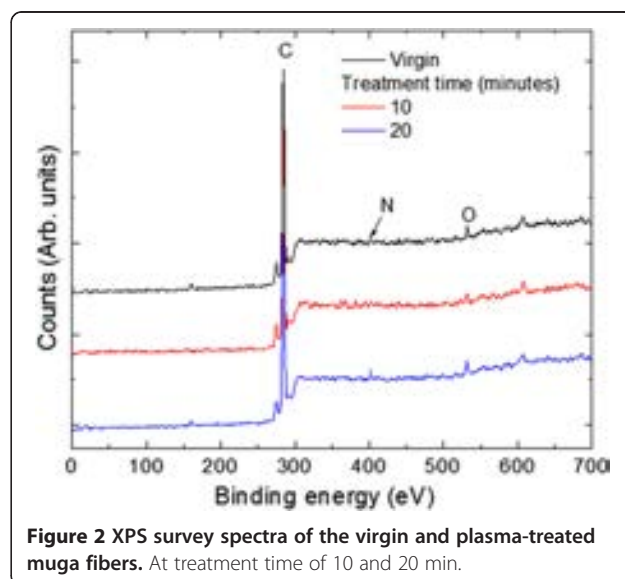


Ala-Ala linkages in the crystalline portion of the silk, whereas the band at  $1,048\text{ cm}^{-1}$  appears due to the presence of the Gly-Ala peptide chain of silk fiber. Moreover, the appearance of the absorption bands at  $3,406$ ,  $2,930$ , and  $1,161\text{ cm}^{-1}$  is caused by the free OH stretching (not hydrogen bonded) and NH stretching (hydrogen bonded) vibrations.  $\text{CH}_4$  group frequency of alanine exhibits an absorption peak at  $1,448\text{ cm}^{-1}$ . The relative abundance of alanine ( $\text{Ala}_n$ ) indicates the existence of alpha phase. From Figure 1, it is apparent that after RF Ar plasma treatment for various times (5 to 20 min), no significant change in the peak position and intensity is observed in the wave number region of  $1,800$  to  $900\text{ cm}^{-1}$  for all the plasma-treated muga silk fibers in comparison to the virgin one. This indicates that in the present investigation, the Ar plasma treatment does not significantly affect the chemical composition of the fibers. However, for the virgin muga fiber, the intensity of the band at  $2,928\text{ cm}^{-1}$  is higher than those of all the plasma-treated muga fibers. This may be attributed to the breakage of some of the H-bonded amide I group due to energetic ion bombardment to the substrates. It is further revealed from Figure 1 that the absorption intensity of the band at  $680\text{ cm}^{-1}$  decreases for plasma-treated muga fibers at a treatment time of 20 min which is possibly accredited to extensive destruction of amide IV and/or  $\text{N-C=O}$  chemical groups present in the fibers by impinging ions, which impart more and more energy to the substrate with higher treatment time.

#### X-ray photoelectron spectroscopy

Figure 2 shows the XPS survey spectra for the virgin and plasma-treated fibers at treatment time of 10 and 20 min.

The variation in atomic concentration, oxygen to carbon (O/C) ratio, and nitrogen to carbon (N/C) ratio of the virgin as well as the plasma-treated fibers is shown in Table 2. For lower treatment time (5 to 10 min), the treated fibers show relatively higher carbon content and lower oxygen and nitrogen contents, as compared to the virgin and other plasma-treated fibers. Above the treatment time of 10 min, the fibers show a gradual decrease in carbon content and increase in both oxygen and nitrogen contents. This is also revealed from the increase in O/C and N/C ratios in the fibers at a higher treatment time of 15 to 20 min. From this finding, it is apparent that with increasing treatment time from 5 to 10 min, the energy of the impinging ions is responsible for the cleavage of peptide chain as well as the breakdown of side chain groups of amino acid, mainly glycine and alanine, which are the major constituents of muga fiber. The breakage of the amino acid groups and peptide chain scission may contribute to the removal of the loosely bonded fibroin region through the formation of various volatile products ( $\text{CO}$ ,  $\text{OH}$ ,  $\text{CO}_2$ ,  $\text{N}_2$ , etc.) which subsequently leads to a decrease in oxygen and nitrogen contents in the fibers. For the treatment time of more than 10 min, the prolonged ion bombardment to the substrate creates severe peptide chain scission as well as the breakage of the amino acid groups, and this possibly lowers the carbon content in the fibers through the formation of more volatile products. Besides, it may lead to the formation of free radicals at the surfaces of the fibers, and on exposure to the atmosphere, these free radicals readily react with atmospheric oxygen/nitrogen and/or water vapor, thereby increasing oxygen and nitrogen contents in the fibers.



**Table 2 Variation in atomic concentration, O/C ratio, and N/C ratio of virgin and plasma-treated muga fibers**

Treatment time (min)	Atomic concentration (%)			N/C	O/C
	Carbon (C)	Nitrogen (N)	Oxygen (O)		
0	91.0	3.3	5.7	0.036	0.062
5	92.5	2.4	4.2	0.025	0.045
10	94.0	1.9	4.1	0.020	0.043
15	89.4	3.7	6.8	0.041	0.076
20	88.5	3.8	7.8	0.042	0.088

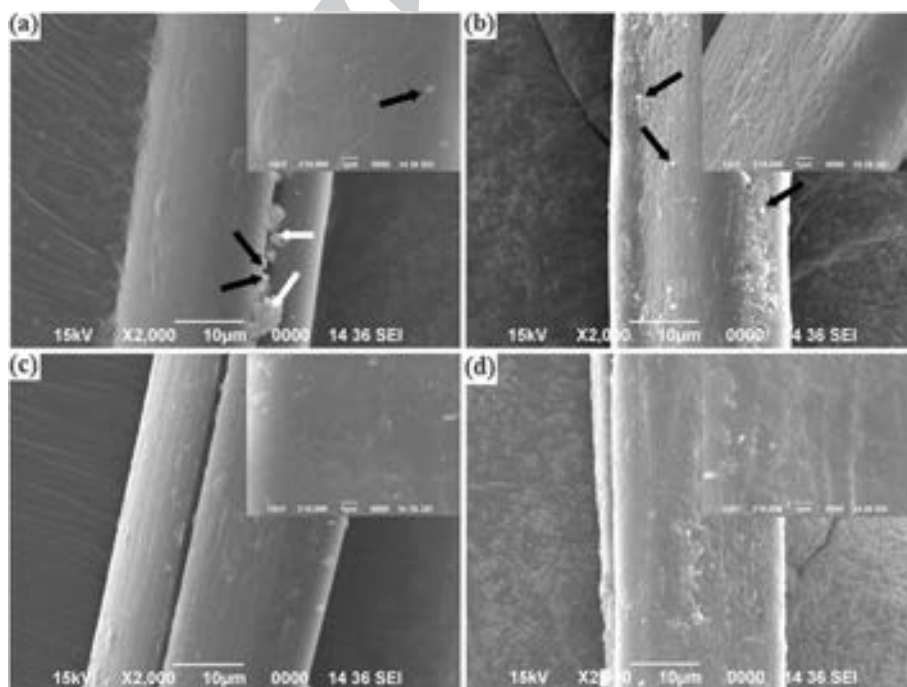
### Scanning electron microscopy

The SEM micrographs of the virgin and Ar plasma-treated muga fibers for 5, 10, and 20 min are shown in Figure 3a,b,c,d. Figure 3a shows the smooth surface of the virgin muga fiber with fewer defects. For treatment time of 5 min (Figure 3b), surface roughness is introduced in the fiber surface due to the ion sputtering effect on the substrates. The fiber treated for 10 min (Figure 3c) shows a relatively smooth surface texture as compared to that treated for 5 min. This is possibly due to the fact that the impinging energetic ions act more efficiently in removing the weakly bonded fibroin regions on the fiber surface. With further increase in treatment time (15 to 20 min), more surface degradation in the form of micro-cracks and voids occur on the fiber surface. The observed crystals on the fiber surface are

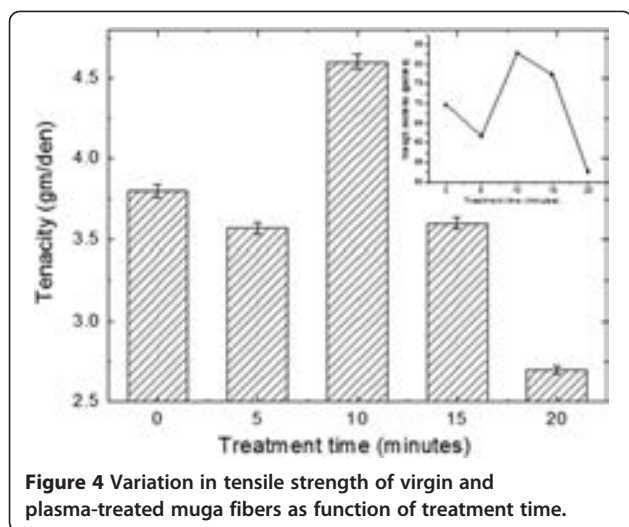
attributed to calcium oxalate deposits which are generally left by the silkworm during spinning as excrements. It is important to observe from Figure 3a that the chemical degumming process is not efficient enough to remove sericin from the innermost gap between the two fibroins. However, as revealed from Figure 3b,d, the treated fibers show less sericin present in the gap between the fibroin. It is therefore apparent that RF Ar plasma treatment on muga fibers can be considered as an efficient and dry degumming process over the conventional chemical process.

### Tensile strength

The variation in tensile strength of the virgin and Ar plasma-treated muga fibers as a function of treatment time is shown in Figure 4. The inset shows the variation in Young's modulus of the fibers as a function of treatment time. It is observed from Figure 4 that at a lower treatment time of 5 min, the tenacity of the Ar plasma-treated muga fibers slightly decreases as compared to that of the virgin one and then it increases to a maximum value for a treatment time of 10 min. Moreover the presence of higher carbon content and also the functional units C-O/C-O-C, C-C/C-H/C=N, and OCOO on the fiber treated for 10 min may further contribute to its enhanced tensile strength. With further increase in treatment time up to 20 min, nearly 41% decrease in



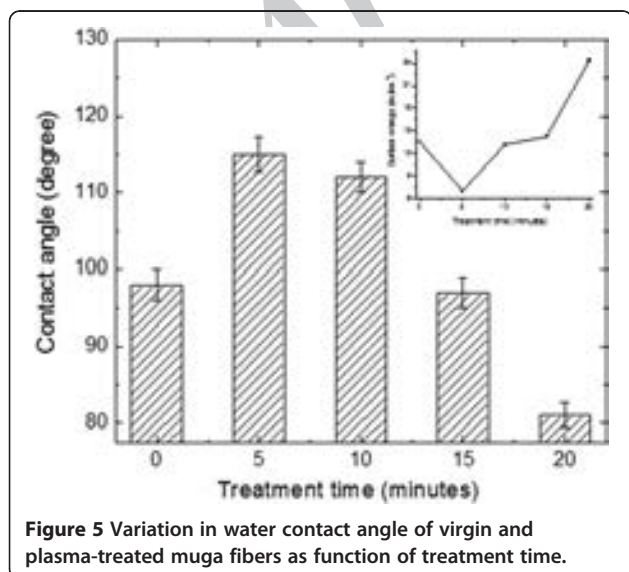
**Figure 3 SEM images.** (a) Virgin and (b-d) plasma-treated muga fibers at treatment time of (b) 5, (c) 10, and (d) 20 min. The black arrows indicate the presence of calcium oxalate, while the white arrow shows the presence of sericin on the surfaces of virgin and plasma-treated fibers. The insets represent the SEM images at higher magnification.



tensile strength is observed as compared to the fiber treated for 10 min. The decrease in carbon content and C-O/C-O-C and C-C/C-H/C=N units is the possible reason for the lowering tensile strength of the fibers treated for longer times (15 to 20 min).

#### Water contact angle measurement

The variation in water contact angle of the virgin and plasma-treated muga fibers as a function of treatment time is shown in Figure 5. The inset shows the variation in surface energy of the virgin and plasma-treated muga fibers as a function of treatment time. The water contact angle of the virgin muga fiber is measured to be 96° (surface free energy (SE) = 12.5 mJ/m<sup>2</sup>). A maximum water contact angle of 115° (SE = 7.0 mJ/m<sup>2</sup>) is observed for the muga fiber treated for 5 min. The fibers treated for 5 to 10 min exhibit enhanced hydrophobicity as compared



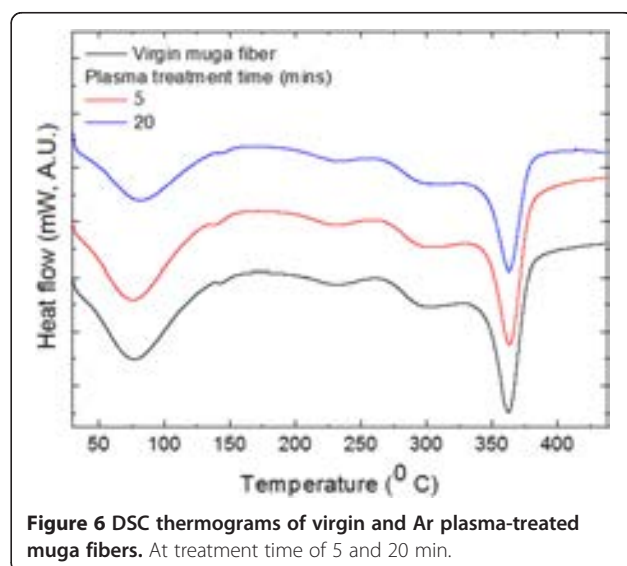
to the virgin one, and this is attributed to the presence of a higher percentage of carbon content and C-C/C-H/C=N, C-O/C-O-C, and OCOO units in the treated fibers. Besides, the lowering in oxygen and nitrogen contents may also contribute to the improved hydrophobicity of the fibers treated for lower times. Further increase in treatment time from 15 to 20 min results in the lowering of the contact angle from 97° (SE = 14.2 mJ/m<sup>2</sup>) to 81° (SE = 24.3 mJ/m<sup>2</sup>), thereby indicating more growth in surface roughness due to prolonged energetic ion bombardment to the substrates which is well revealed from the SEM micrographs (Figure 3d). The decrease in water contact angle on the fiber surface at higher treatment time can further be correlated with the decrease in carbon content and C-C/C-H/C=N units in the fibers. An increase in oxygen content and the O=C-N/C=O/O-C-O and OCOO units also apparently contribute to the poor hydrophobicity of the fibers subjected to longer plasma treatment time.

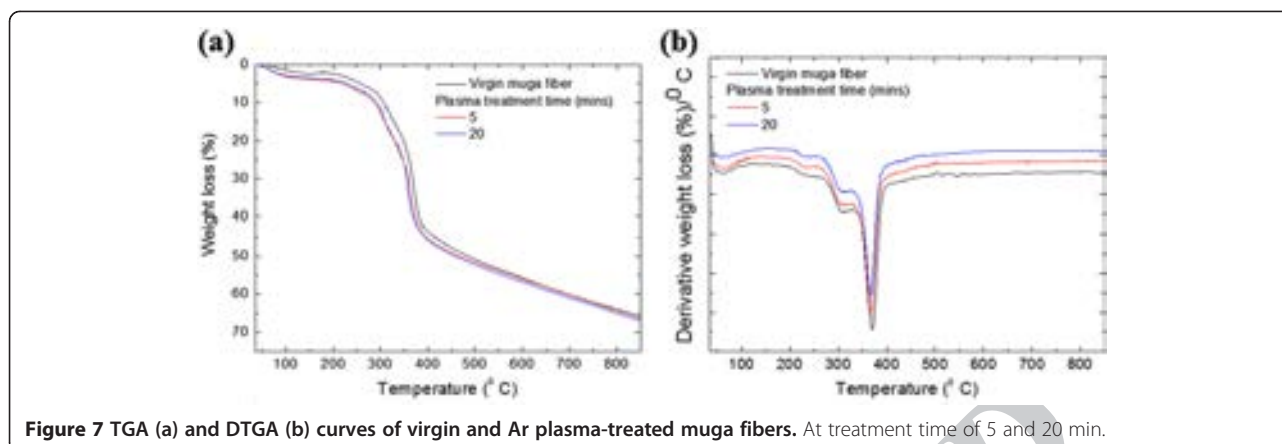
#### Thermal stability

The thermal behavior of virgin and plasma-treated muga silk is studied with the help of DSC and thermogravimetric analysis (TGA) as represented in Figures 6 and 7. From the presented results, it is revealed that there is no major change in thermal stability of muga silk due to plasma treatment. It has an important significance in this work that the bulk properties of muga silk are not significantly altered except surface properties.

#### Conclusions

Ar plasma treatment on muga fibers has been carried out at an RF power of 20 W and treatment time range of 5 to 20 min. From FT-IR studies, it is revealed that Ar plasma-treated fibers exhibit a chemical behavior





**Figure 7** TGA (a) and DTGA (b) curves of virgin and Ar plasma-treated muga fibers. At treatment time of 5 and 20 min.

similar to that of the virgin fiber. At treatment time of 5 to 10 min the plasma-treated fibers show improved tensile strength and hydrophobicity as compared to the virgin one. The presence of higher carbon content and C-O/C-O-C, C-C/C-H/C=N, and OCOO units may further be accounted for the improvement in tensile strength and hydrophobicity of the fibers treated at lower treatment times. The reduction in carbon content and incorporation of more oxygen-containing functional groups at higher treatment times (15 to 20 min) are other important factors which decrease the properties of the fibers. The results suggest that RF Ar plasma treatment on this muga silk fiber is an innovative way to upgrade its qualities only by altering its surface chemistry and hence to successfully use as biomaterials such as muga silk suture.

## Methods

### Materials

Muga silk fibers used in this research work are provided by the Regional Muga Research Station, Central Silk Board, Mirza, Government of India. The silk fibers are cleaned in alkaline solution to remove sericin before plasma treatment, repeatedly washed with distilled water, and dried in air atmosphere (temperature 27°C and relative humidity 65%). The fibers are cut into a length of 6 cm and are held stretched on a horizontal stainless steel plate with the aid of holding rings providing a treatment area of 36 cm<sup>2</sup>.

### Plasma treatment

The plasma treatment process is carried out in a vertically placed stainless-steel cylindrical chamber of 40 cm in diameter and 45 cm in length. The chamber is evacuated to a base pressure of  $1 \times 10^{-3}$  mbar using a rotary pump (21 m<sup>3</sup> h<sup>-1</sup>). Argon gas (purity > 99.9%) is fed in the chamber through a flat cylindrical gas shower plate (9.25 cm in diameter). The bottom surface of the shower plate contains 60 holes, each having a diameter

of  $5 \times 10^{-2}$  cm. The RF electrode (10 cm in diameter) is placed horizontally at the center of the chamber with proper insulation and connected to an RF (13.56 MHz) source (COMDEL-CPS-500 AS, 0 to 500 W, Gloucester, MA, USA) through an L-type matching network. Cold water is circulated through the electrode so as to keep it cool during the plasma treatment process. The gas shower plate is placed 5 cm above the RF electrode, and the samples are placed on the surface of the electrode. The Ar plasma treatment is carried out at an RF power of 20 W and treatment time range of 5 to 20 min. The argon gas flow rate is set at 5 sccm (MFC) (Aalborg, Orangeburg, NY, USA) during the whole plasma treatment process. Finally after treatment, the RF power is turned off, and the substrates are allowed to cool in an argon flow (35 sccm) for 15 min. The samples are collected from the RF chamber after 6 h and immediately transferred to a vacuum desiccator.

During RF Ar plasma treatment, measurement of the plasma discharge parameters such as electron density, electron temperature, and plasma potential is carried out using self-compensated planar Langmuir and emissive probes with the same type of compensating circuit. The self-compensated circuit is used to minimize the perturbation effect of the RF voltage across the probe sheath [12-14]. The electron temperature and the ion and electron densities are evaluated from the current-voltage (*I-V*) characteristics obtained using the self-compensated Langmuir probe, while the plasma potential is estimated using the self-compensated emissive probe. The plasma potential is evaluated from the *I-V* characteristics obtained with the emissive probe using the inflection point method. During all measurements reported here, both probe tips are kept 3 cm above the RF electrode. The OES technique is used to obtain information about the excited Ar spectral lines during the plasma discharge. For this, a 300-mm-focal-length monochromator (ANDOR, Belfast, UK), having a resolution of 0.1 nm and equipped with a 1,200-line/mm

grating, is used (wavelength accuracy =  $\pm 0.2$  nm). The Ar spectral lines are studied in a wavelength range of 300 to 900 nm. A fiber-optic probe is put approximately 15 cm away from the plasma discharge. Proper care is taken to detect maximum light emission and avoid background radiation.

### Characterization

The chemical compositions of the virgin and Ar plasma-treated muga fibers are studied by Fourier transform infrared spectroscopy (FT-IR, Bruker Vector 22, Madison, WI, USA). The spectra are obtained in the transmittance mode, within the spectral range of 4,000 to 400  $\text{cm}^{-1}$ . All FT-IR measurements are performed with 32 scans and at a resolution of 4  $\text{cm}^{-1}$ .

The surface chemistry of the virgin and Ar plasma-treated fibers is investigated by X-ray photoelectron spectroscopy (XPS). The XPS studies are conducted in a UHV chamber (base pressure  $< 2 \times 10^{-8}$  mbar) using a VG-made CLAM-2 model hemispherical analyzer (East Grinstead, UK) with a non-monochromatic twin Mg X-ray source with the emission voltage and current of the source set to 15 kV and 16 mA, respectively. With a  $\text{MgK}_{\alpha}$  line (1,253.6 eV, passing energy = 23.5 eV), detailed spectra are collected, followed by a high-resolution scan of relevant core-level and valence-level photoemission peaks of all the main elements. The XPS curve fitting is performed and analyzed using the XPSPEAK 4.1 software.

The effect of Ar plasma treatment on the crystallinity of the muga silk fibers is investigated by using an X-ray diffractometer (D8 ADVANCE, BRUKER, Karlsruhe, Germany) at a scan speed of 0.1 s/step with a step angle of  $0.5^{\circ}$  by symmetrical reflection geometry in a  $2\theta$  range of  $10^{\circ}$  to  $40^{\circ}$ . The X-ray line used is nickel-filtered  $\text{CuK}_{\alpha}$  radiation, and a curved crystal graphite monochromator is used before the detector.

Scanning electron microscopy (SEM; JEOL-6390 LV, Akishima-shi, Japan) is performed to study the surface morphologies of both virgin and plasma-treated muga fibers. The samples are coated with platinum in an ion-sputter coater (JFC 100, JEOL) in a low vacuum prior to examination.

Atomic force microscopy images are obtained in ambient conditions with a digital multimode scanning probe microscope (Veeco, Plainview, NY, USA) equipped with a nanoscope IV A controller (resolution: horizontal, 0.2 nm and vertical, 0.01 nm). The images are taken in tapping mode at a scanning rate of 1 Hz. The rms value of the surface roughness of the virgin and Ar plasma-treated muga fibers is measured using a scanning probe microscopy software (WSxM).

The tensile strength of the virgin and treated fibers is measured using an Instron tensile tester (INSTRON

4204, Norwood, MA, USA) at a constant speed of 10  $\text{mm}/\text{min}^{-1}$  with a 10-N load (room temperature  $27^{\circ}\text{C}$  and relative humidity 65%). All measurements are repeated for five equally treated samples to obtain average values of tenacity.

The dynamic contact angles of the virgin and plasma-treated muga fibers are measured using a tensiometer (DCAT-11, Data Physics, San Jose, CA, USA) at ambient condition. The instrument provides a measuring contact angle accuracy of  $\pm 0.01^{\circ}$  with a position resolution of 0.1  $\mu\text{m}$ . The fiber is immersed into the liquid up to a depth of 1.5 mm with the help of a computer-controlled motor (speed 0.1 mm/s). The contact angles on the fibers are determined using the Wilhelmy technique. The surface free energy of the virgin and Ar plasma-treated fibers is evaluated by the universal Owens-Wendt-Rabel-Kaelble method.

The differential scanning calorimetry (DSC) thermograms are recorded with a PerkinElmer thermal analyzer (DSC-6000, Waltham, MA, USA; temperature accuracy 0.25%, weighing precision 0.01%) coupled with thermoanalyzer processor. The thermal degradation of the muga fibers is studied using a PerkinElmer thermal analyzer (TGA-4000; temperature accuracy 0.25%, weighing precision 0.01%). The samples (2 to 3 mg) are heated in nitrogen atmosphere (flow rate 20 ml/min), at a heating rate of  $10^{\circ}\text{C}/\text{min}$ , over a temperature range of  $30^{\circ}\text{C}$  to  $850^{\circ}\text{C}$ .

### Competing interests

The authors declare that they have no competing interests.

### Authors' contributions

DG carried out the complete experimental work. Prof. JC directed the whole work during the complete experiment. Dr. AJC carried out the analytical and the sample characterization part. Dr. ARP helped design the experimental setup as per the requirement. Dr. DP helped correlate the complete results. All authors read and approved the final manuscript.

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