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Influence of UV radiation on the viscoelastic properties and dynamic viscosity of bovine hide using dynamic mechanical analysis

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Abstract The wide range of applications of collagen-based materials has triggered research interest especially on the effect of environmental factors in that these materials are exposed to during processing and application. As the applications of these collagenous materials continue to increase such as in the field of medicine, more studies are required to gain more insight into their properties. Collagen is a natural biopolymer whose structure is sensitive to ultraviolet (UV) radiations, which alters its mechanical properties. In this study, the influence of artificial UV irradiations, wavelength 254 nm, on the viscoelastic properties and dynamic viscosity of both pickled and tanned hide was investigated by dynamic mechanical analysis. The influence of tanning on the viscoelastic properties and dynamic viscosity was also investigated. Freshly flayed bovine hide was conventionally prepared to pickling stage and split into two identical halves along the backline. One half was tanned using chromium sulfate, while the other half was left at the pickled stage. Samples of appropriate dimensions from both the pickled and tanned hides were cut and irradiated with artificial UV light for different time duration of 6–30 h. The irradiated samples were then analyzed using the DMA in the multi-frequency mode. It was found that irradiation caused an increase in the storage modulus (E') of pickled hide over the entire irradiation of 6 h followed by consistent decrease up to a duration of 30 h. Tanning caused an increase in tanδ that consistently decreased with the increase in the duration of irradiation. In addition, UV irradiation caused an increase in dynamic viscosity of pickled hide, but a decrease in tanned hide. The results show the predominant elastic nature of bovine hides as indicated by tanδ magnitudes less than a unit.

Keywords Thermal and dynamic mechanical analysis · Bovine hide · UV radiation · Viscoelastic properties · Dynamic viscosity · Collagen

Introduction

Bovine hide forms an important raw material for many industries, and its applications span a wide range of fields such as clothing, tannery, medicine, biotechnology, food industry, among others. The main component of this hide is collagen, which is a natural polypeptide biopolymer that confers mechanical properties to the hide [1, 2]. Among the 26 types of collagens known, the main structural component of animal hide and skin is type I that forms the basic strong fibril [3]. The collagen triple helix of collagen type I is sensitive to UV-254 nm radiation [4]. When this collagen is exposed to these UV radiations, photopolymerization in the telopeptide regions of the molecule occurs and some energy causes local heating [4–6]. The radiations absorbed by the collagen alter both its physical and chemical structures [7]. The UV radiation absorption is enhanced by the presence of aromatic groups such as tyrosine and phenylalanine. The photodegradation of these residues creates free radicals that either recombine to form crosslinks or undergo chain scission [7–9].

Owing to the growing applications of this biomaterial and the increasing use of radiation in the fundamental industrial processing of these materials, the investigation of

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the effects of the radiation and vulnerability of these collagenous material to UV radiation was necessary [10–12].

The viscoelastic properties of these collagenous materials have been studied [13–15]. Further, the influence of UV radiation on collagen has also been investigated [7, 12, 16, 17]. However, the effect of UV radiations on the viscoelastic properties and dynamic viscosity of bovine hide has not been done to the best of our knowledge. This study therefore investigated the effect of artificial UV radiations on the storage modulus, tanδ and dynamic viscosity of both pickled and tanned bovine hides using dynamic mechanical analysis.

Materials and methods

Sample preparation

A freshly flayed bovine hide was commercially procured and preserved by use of sodium chloride and a fungicide, Mirecdide TC 1080 V, for 2 h to restrict bacterial and fungal activities. The hide was prepared to the pickled condition by conventional process. The pelt was cut into two identical halves along the backline. One piece was tanned using chromium sulfate and neutralized in sodium bicarbonate. Rectangular specimens of dimensions $30\,\text{mm} \times 9.3\,\text{mm} \times 0.93\,\text{mm}$ sampled according to the official sampling method and sampling location ISO 2418:2002 were cut using a press knife. The press knife cut out the specimen such that the angle formed at the cutting edge between the internal and external surfaces of the press knife was about 20 degrees and the depth of the wedge of the cutting knife was greater than the thickness of the cut leather, as illustrated in [18].

UV irradiation

The samples were irradiated in air at room temperature and at room relative humidity with UV light of wavelength 254 nm (UV-C) and irradiance of 0.0475 Wm^{-2} from a 8-W UV fluorescent lamp (Model LF-204.LS) from UVITEC. The lamp was positioned horizontally 15 cm away from the sample in an aluminum shutter. Various doses of UV irradiation were achieved by varying duration of irradiation from 0 h (no irradiation) to 30 h. The samples were then conditioned in a standard atmosphere 23/50 (temperature $T = 23 \pm 2$ °C, humidity $\phi = 50 \pm 5$ % R.H.) for at least 48 h according to ISO 2419:2002 (IULTCS/IUP 3) standards prior to testing.

Dynamic mechanical analysis

Using the DMA (Model 2980 from TA instruments), a variable-amplitude sinusoidal mechanical stress was applied to each of the pickled and tanned samples to produce a sinusoidal strain of preselected amplitude. The samples were mounted onto the film tension clamp, and the experiment was run in the multi-frequency mode over the frequency range 0.1–100 Hz equilibrated at 30 °C and a heating rate of 5 °C min^{-1} to 270 °C in static air environment. The heating rate chosen was the ideal rate for film tension geometry dictated by the thickness and nature of the sample, according to the DMA 2980 manual. The parameter measured was complex modulus which was resolved into real and imaginary components, the storage modulus (E') and loss modulus (E''), respectively. Storage modulus determines energy stored and is related to the stiffness of the material representing the elastic portion of the viscoelastic material, while loss modulus gives the dissipated energy as heat per given cycle representing the viscous portion or plasticity of leather [19]. The ratio of energy dissipated to energy stored per cycle is given as tanδ, where δ is the phase angle between the stress and strain in the oscillatory experiment and this represents the damping or loss factor. These parameters give important information on the bulk properties related to their functional performance.

Results and discussion

Storage modulus (E′)

The comparison of storage moduli (E') of non-irradiated (control) pickled and tanned hides is illustrated in Fig. 1. Pickled hide showed progressive increase in E' with temperature up to two distinct peaks of 1192 and 1101 MPa at temperatures 110 and 195 °C, respectively. There was a minimum E' value of 820 MPa at 135 °C and a drop to almost zero at 210 °C. Tanned hide showed a peak of 1418 MPa at 225 °C. Minimum E' of 813 and 765 MPa occurred at 40 and 85 °C, respectively. During the tanning, pre-tanning processes and exposure to the atmosphere, collagen matrix absorbs water [20]. Increase in temperature dehydrates the collagen, making the fibrils and bundles to come together and the structure to be more compact and stiff, hence increasing E' [21, 22]. Beyond the first peak, the storage modulus decreases due to the melting of the amorphous fraction of collagen. A further increase in temperature causes the water molecules in collagen to compete for energy with the hydrogen bonds that maintain the triple-helical configuration causing the unwinding of the triple helix (denaturation) and final formation of localized gel in the case of pickled hide [22]. Denaturation leads to a decrease in the storage modulus. This process has also been referred to as melting of the native crystalline (rigid) collagen or shrinkage of the native collagen both denoting glass transition [14, 23]. During this melting,

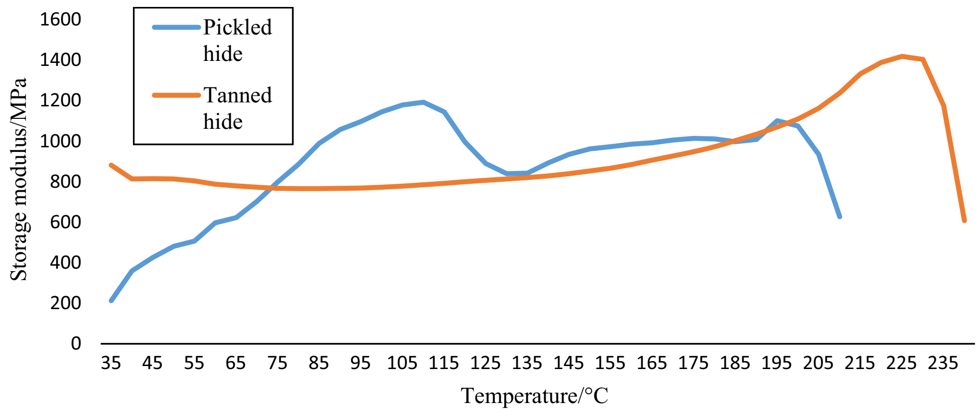


Fig. 1 Storage modulus of non-irradiated pickled and tanned hides

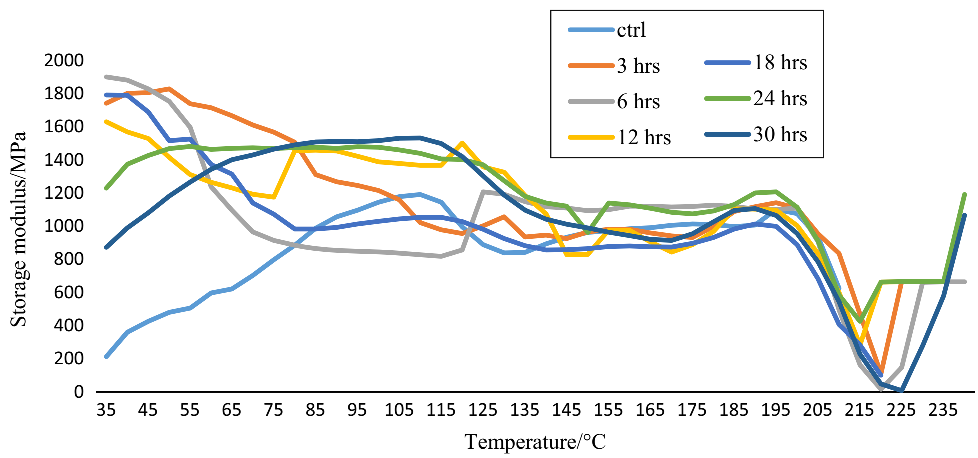


Fig. 2 Effect of UV irradiation on storage modulus of pickled hide

there is increased mobility of the macromolecular chains that weaken the intra- and inter-collagen bonds, hence decreasing the storage modulus.

The influence of UV irradiation on E' of the pickled hide is illustrated in Fig. 2. At temperatures lower than 110 °C, the control sample had the lowest values of E' compared to the irradiated samples. However, there was a characteristic trend such that the sample irradiated for 30 h had the highest E' followed by 24 h, 12 h followed by 18 h then 6 h. Higher irradiation showed significant increases in E'. These results agree with the findings of Liu et al. [24]. Collagen amino acids possess endogenous chromophoric sites (aromatic residues) such as tyrosine and phenylalanine which absorb the UV radiations in the range of 250–300 nm [12, 25–27]. The energetic UV radiations initiate photodegradation of these aromatic amino acids to form crosslinks called dityrosine [12, 17, 26, 28, 29]. This enhances the hardness and stiffness which increases the E' [12, 30]. Further absorption of the UV radiation forms new photoproducts such as pentosidine and pyridinoline that add more crosslinks [9, 16]. However, the concurrent polypeptide chain scission initiated by the UV irradiation causes intermediate magnitudes of E'. Similarly, water molecules in the collagen matrix split under UV irradiation, forming radicals which attack peptide backbone radicals (–NHC–CO–). The radicals recombine, forming covalent bonds together with the subsequent modification of the amine and carboxyl groups' crosslinks and hence increasing the storage modulus [31]. This agrees with results obtained by [31] who attributed the intermediate magnitudes to competing processes of peptide bond scission events arising from the free radical mechanism of the aromatic residues donating free electrons.

The effect of UV radiation on the tanned hide is shown in Fig. 3. The samples irradiated for 30 min, 3, 12 and 30 h had lower storage modulus values than the control sample, while samples irradiated for 6 and 18 h had higher E' than the control. Beyond 30 min, E' increased to the highest value at 6 through 3 h. Thereafter, the storage modulus decreased through 12 and 18 to 30 h. From 0 to 30 min, the decrease in E' indicates the photodegradation of collagen along its main chains with scission of $-CH_2-N=$ and $=CH_2$ bonds. It is likely due to the breaking of N–H···O=C inter-chains hydrogen bond as well as the loss of the bonding water in collagen [32]. Thereafter, the E' starts to increase slowly to 6 h where the magnitude is highest. This can be attributed to the crosslinking as a result of formation of dityrosine, pentosidine and pyridinoline. Further irradiation and increased absorption causes photodegradation and photolysis that weakens the bonds and decreases the storage modulus. Comparing Figs. 4 and 5, the trends are different. Apart from the tyrosine, phenylalanine, pentosidine and pyridinoline that increase UV absorption, the presence of chromium ions acts as synthetic polymer or special inorganic pigment increasing absorption [33, 34]. Similarly, tanning makes the leather structure compact lowering the percentage reflectance and increasing the absorption. This absorption complicates further the polypeptide crosslinking and chain scission. Absorption coefficient is affected by hydration level owing to direct absorption by water molecules and subsequent vaporization [35]. At higher irradiation duration, the maximum of absorption/scattering is almost the same. Pickled hide behaves as undyed crust which has high reflectance of the UV radiations that scatter the radiation, and hence their impact on the structure is minimal [36]. Free radicals appear in collagen water solutions under UV radiation and evoke photodegradation of the macromolecule [37].

Fig. 3 Effect of UV irradiation on storage modulus of tanned hide

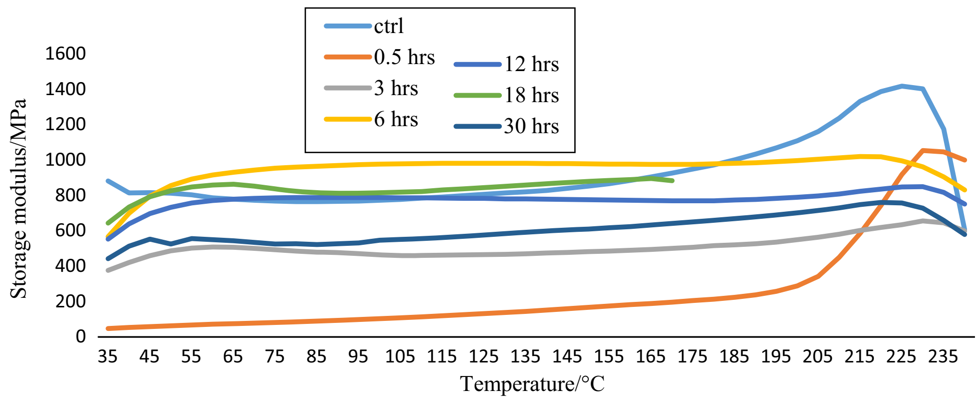


Fig. 4 Effect of tanning on tanδ of pickled and tanned hides

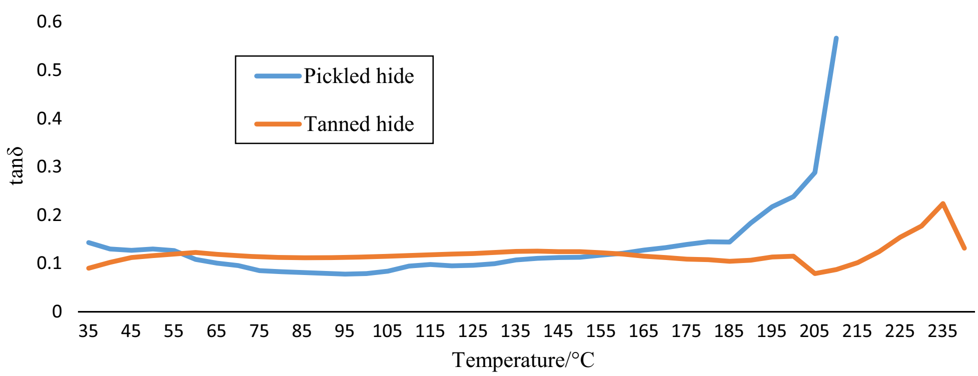
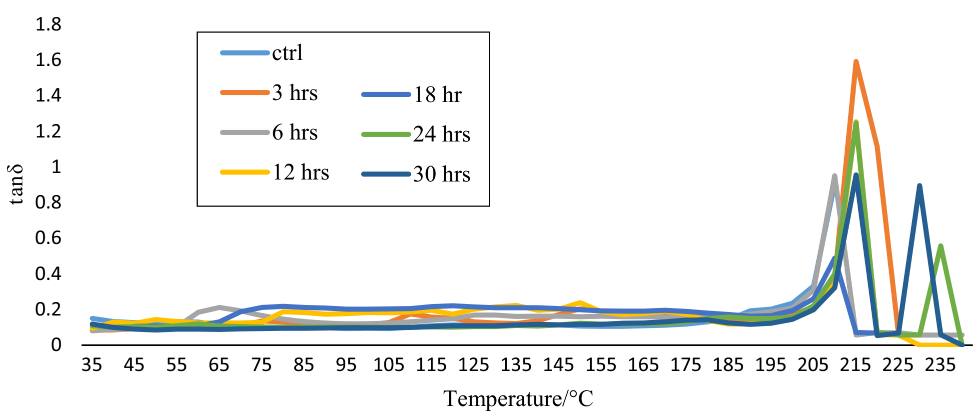


Fig. 5 tanδ of pickled hide at different irradiation durations



tan delta (tanδ)

Figure 4 illustrates the influence of tanning on the tanδ. Both pickled and tanned hides possess some damping capability useful to dissipate cyclic mechanical energy imposed on it during deformation. As it can be seen in the figure, in the temperature range 55–165 °C, tanning slightly increases the dissipative capability of the collagen fibers as indicated by slightly higher tanδ of tanned hide compared to pickled hide. However, this damping is higher in pickled hide than in tanned hide at temperatures higher than 165 °C.

Figure 5 illustrates the effect of UV irradiation on tanδ of pickled hide. Although there was overlapping in tanδ due to smaller values in the temperature range 35–210 °C, there appears a characteristic trend. The tanδ was highest in the specimen irradiated for 18 h, and then it decreased in the order 12, 6, 3 h then control. In the temperature range 35–210 °C, the magnitudes of tanδ were <0.5, implying the elastic nature of the hide. tanδ increased rapidly higher than 210 °C, although only samples irradiated for 12 and 18 h recorded tanδ greater than a unit. This implies that pickled hide is predominantly elastic in nature at these temperatures. The viscous component is only noticed at higher temperature when the mobility of the peptide chains associated with the dissipation of energy in form of heat being high [38].

The influence of UV irradiation on tanδ in tanned hide is illustrated in Fig. 6. For all the samples, tanδ increased rapidly at temperatures greater than 180 °C. This is due to increased chain mobility that increases the energy dissipation in the form of heat.

Dynamic viscosity

Figure 7 illustrates the effect of tanning on the dynamic viscosity of hide. The dynamic viscosity increased in both hides with temperature forming peaks at different temperatures. The initial increase in dynamic viscosity with temperature is possibly due to the increasing hydrophobic interactions between the collagen molecules as collagens associate more at high temperatures [39]. With increasing temperature, energy for heat motion of the polypeptide chains increases and thus the resistance to segment motion becomes weaker, leading to drop in dynamic viscosity [40]. Similarly, during melting or denaturation of the collagen,

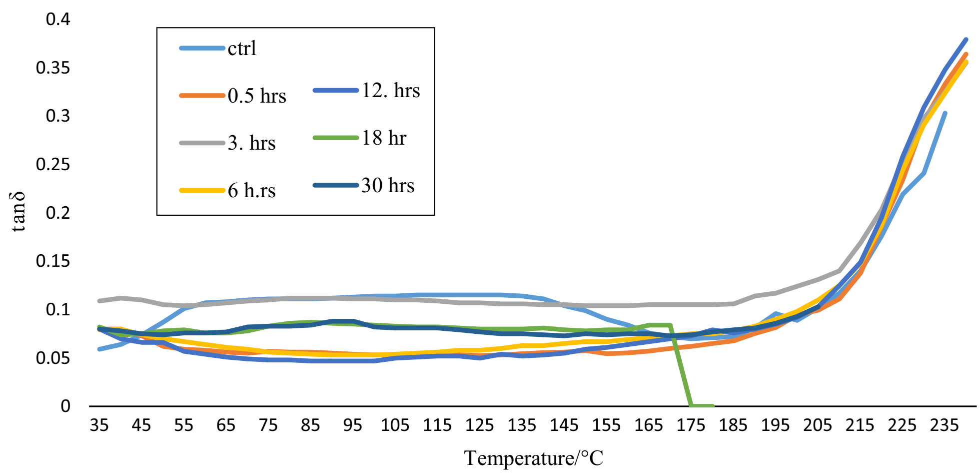
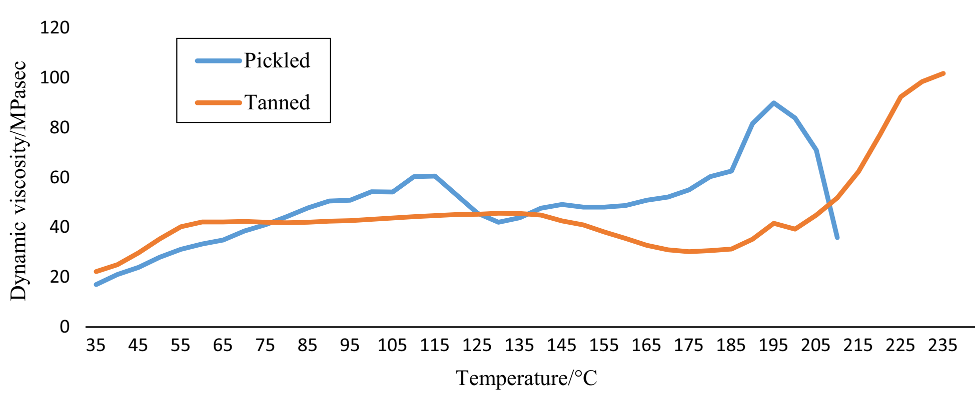


Fig. 6 Effect of UV irradiation on tanδ of tanned hide

Fig. 7 Influence of tanning on dynamic viscosity of hide



the triple helix of collagen collapse to a random coil with weak intra- and inter-molecular hydrogen bonding that holds the secondary structure of collagen [41–43]. This leads to a decrease in the dynamic viscosity. At temperatures higher than 195 °C, the dynamic viscosity of pickled dropped continuously, while it increased rapidly in tanned hide. This is probably due to the excess tannins that generate additional crosslinks of collagen fibrils leading to reinforcement of the tanned hide at higher temperatures [44]. The dynamic viscosity curves peaks in pickled hide were sharper than those observed in tanned hide. Major peaks in pickled hide were at 115 and 195 °C of magnitudes 60 and 90 MPasec, respectively, and a minimum of 45.65 MPasec at 130 °C, while at 135 °C of 45.55 MPasec in tanned hide. Dynamic viscosity of pickled hide was greater than that of tanned hide in the temperature range 75–205 °C. The collagen spaces created by the swelling processes in tanned hide were filled up by the chromium ions. This leaves little space for water absorption, and hence the increases and the peaks in tanned hide were less sharp and smaller.

Figure 8 illustrates influence of UV irradiation on dynamic viscosity of pickled hide. The control sample had the lowest dynamic viscosity followed by the sample irradiated for 30 h with the specimen irradiated for 30 min having the highest dynamic viscosity with a minimum of 439 MPasec and a maximum of 949 MPasec at 185 and 100 °C, respectively. All the specimens showed a distinct common trend, whereby the dynamic viscosity reached maximum at 90–130 °C.

The effect of UV irradiation on the dynamic viscosity of tanned hide is illustrated in Fig. 9.

The control sample had the highest dynamic viscosity than the irradiated samples. This implies that dynamic viscosity decreases with irradiation. This agrees with results from collagen studied by Sionkowska [25] and Sionkowska et al. [16]. In both investigations, the relative viscosity decreased with time of UV irradiation. The results also agree with the viscosity measurements by Fathima et al. [9] on both native and aldehydes-treated collagens. The reduction in dynamic viscosity can be attributed to the cleavage of peptide bonds of the rod-like triple-helical molecule [28]. The presence of chromium ions in the collagen matrix plays the role of synthetic polymer that increases the absorption of UV irradiation as observed in a study by Sionkowska [34]. Increased absorption causes

Fig. 8 Influence of UV irradiation on dynamic viscosity of pickled hide

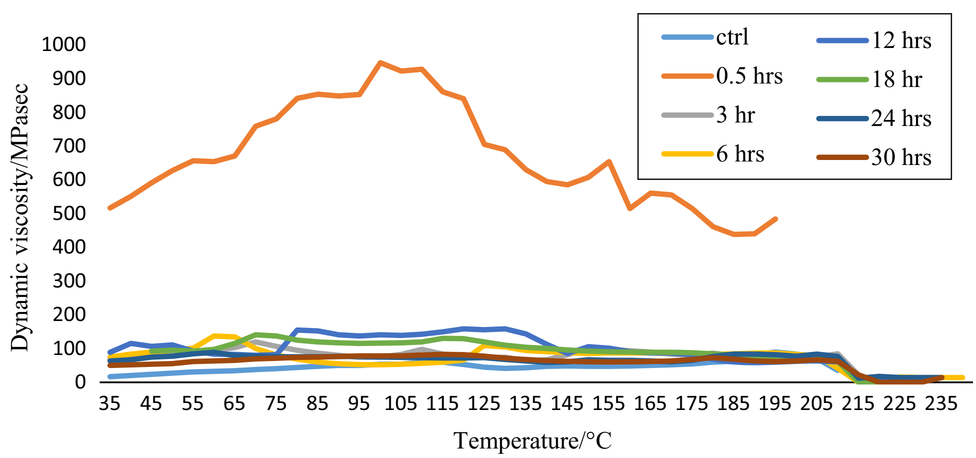
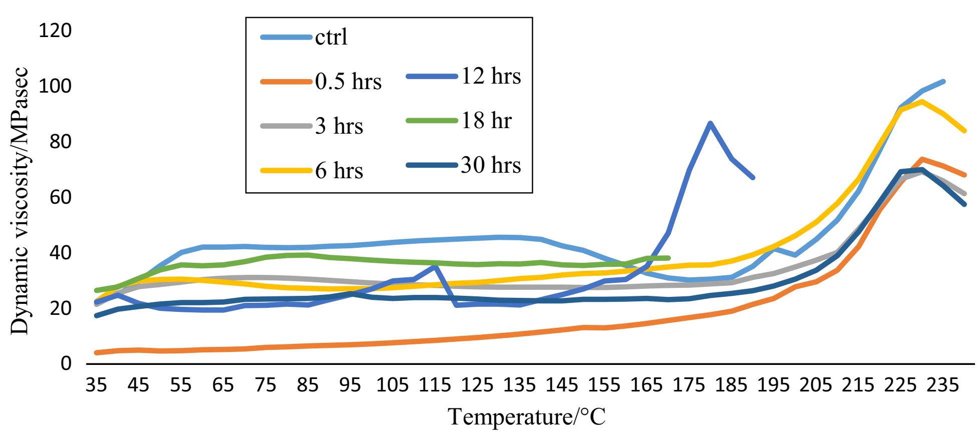


Fig. 9 Influence of UV irradiation on dynamic Vvscosity of tanned hide



major photodegradation and scission in the main chains and hence weak stability that leads to decreased dynamic viscosity for irradiated samples [9]. Similarly, the distinct bluish color of the tanned hide is good for the absorption of UV irradiations than the highly reflecting undyed pickled crust that scatters leading to low impact on its structure [35]. These results significantly correlate with those of Olle et al. [45] whereby the chrome-tanned leather was strongly affected by the UV radiations than the wet white leather.

Conclusions

It was found that irradiation increases the storage modulus (E') of pickled hide, whereas in tanned hide, irradiation increased E' up to maximum at 6 h followed by the consistent decrease with irradiation until 30 h of irradiation. It was observed that tanning increases the damping capability as indicated by higher tanδ in tanned than in pickled hides. The results also show the predominant elastic nature of hides. In pickled hide, tanδ consistently decreased with irradiation. Further, it was found that irradiation increases the dynamic viscosity of pickled hide, but decreases that of tanned hide.

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