

Determination of Surface Properties of Human Hair by Inverse Gas Chromatography and Dynamic Vapour Sorption

DVS/iGC SEA Case Study 616

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This paper describes the application of Inverse Gas Chromatography Surface Energy Analyzer (iGC SEA) to determine the surface energetic heterogeneity of three human hair samples; caucasian virgin (undamaged), thermally damaged (70°C) and chemically damaged (bleaching) hairs. Dynamic Vapour Sorption (DVS) was used to investigate the water sorption performance of the hair samples

Introduction

Bleaching and exposure to high temperatures or light can damage the inner structure of the hair and cause detectable changes in the surface properties. For example, bleaching agents and exposure to light cause the oxidative destruction of the pigments in the inner structure of the hair [1], and the surface of the hair is also affected by these treatments. Therefore, various pretreatments cause detectable changes on the surface. Other important components in hair include amino acids, which cause the slightly acidic nature of the hair surface. For these reasons, understanding and measuring surface properties of hair fibres has drawn interest in the personal care industry.

The use of dynamic methods such as Inverse Gas Chromatography Surface Energy Analyzer (iGC SEA) and Dynamic Vapour Sorption (DVS) allows accurate determination of the vapour sorption capacity as well as surface energy. In particular, IGC can be used to determine the dispersive component of surface energy and the interaction with polar probes can be used to study acid-base properties.

Method

Three types of human scalp hair samples; caucasian virgin (undamaged), thermally damaged (70°C) and chemically damaged (bleaching) were used. The hair samples were taken from a few centimeters above the ends to avoid sampling the ends themselves as they may be damaged more severely. For the DVS experiments a typical partial profile of 0 – 95% RH in 10% steps at 25 °C was used. A DVS-Advantage (SMS, Alperton, UK) instrument was used for this study.

In the case of the IGC SEA experiments each standard hair sample was packed in a column with 30 cm length and a 4 mm ID. All IGC analyses were carried out using iGC Surface Energy Analyzer (SMS, Alperton, UK) and the data were analysed using both standard and advanced SEA Analysis Software. Samples were run at a series of surface coverages with alkanes and polar probe



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molecules to determine the dispersive surface energy distribution as well as the acid-base surface energy distribution. For the analysis, the Dorris and Gray method was employed for the dispersive component [2]. The acid-base contribution was determined by first measuring the free energy of desorption based on the polarisation approach [3]. The specific surface energies were determined based on the van Oss approach and Della Volpe scale [4]. Each column was pre-conditioned for 2 hours at 30°C and 0% RH with helium carrier gas to remove any physisorbed water. All experiments were carried out at 30°C with 10sccm total flow rate of helium, using methane for dead volume corrections.

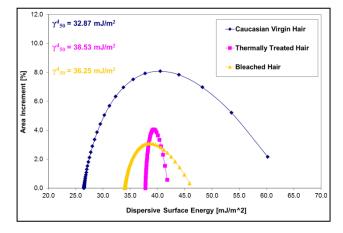
Results

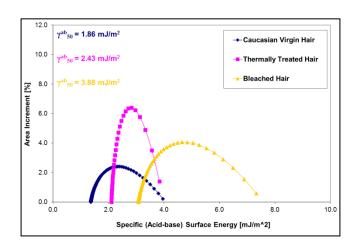
Surface Energy Heterogeneity

Dispersive, γ_S^D and specific (acid-base), γ_S^{AB} surface energy profiles obtained directly from the iGC SEA for caucasian virgin (undamaged), thermally damaged (70°C) and chemically damaged (bleaching) samples are shown in Figure 1 and Figure 2, respectively.

The results show the energetic changes on the hair samples due to different damaging treatments. From Figure 1, undamaged caucasian virgin hair sample was energetically heterogeneous with a mean dispersive surface energy, γ_s^D of 32.87 mJ/m². Heat treatment and bleaching causes a shift to higher mean γ_{S}^{D} values $(38.53 \text{ mJ/m}^2 \text{ and } 36.25 \text{ mJ/m}^2, \text{ respectively}),$ indicating an increase in the number of high energy sites on the surface of hair. This might be due to the low energy lipid layer which covers the cuticle cells of hair being removed. Grooming procedures may have damaged the surface of these cells, therefore eliminating the lipid layer. As such treatments have created a high degree of cuticle damage, polar probe molecules were able to reveal more significant differences between the hair samples.

From Figure 2 the rank order of increasing specific interactions, γ_{S}^{AB} can be categorised as caucasian virgin (1.86 mJ/m²), thermally damaged hair (2.43 mJ/m^2) and bleached damaged hair (3.88 mJ/m^2). From Figure 3 the samples show the following trend with free energy values: acetonitrile > acetone > dichloromethane > ethyl acetate, indicating the amphoteric nature of the hair samples. The increased free energy values for the bleached hair sample may be indicative of more active polar surface sites (higher energy surface groups, or higher density of active surface groups), which in turn increase the wettability. Figure 4 shows the wettability (also known as hydrophilicity) profiles for the samples, which can be determined by dividing specific surface energy by the total surface energy ($\gamma_{s}^{ab}/\gamma_{s}^{t}$).





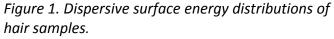




Figure 2. Specific (acid-base) surface energy distributions of hair samples.

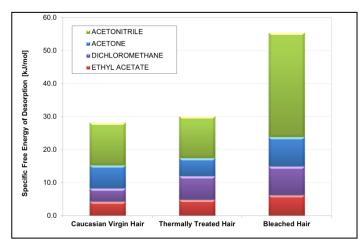


Figure 3. Specific free energies of desorption values for the samples (values grouped by sample).

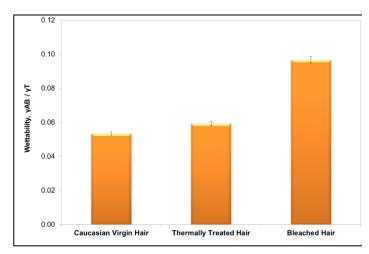


Figure 4. Wettability, $\gamma_{S}^{AB}/\gamma_{S}^{T}$ profiles for the hair samples.

Water Sorption Measurements

The water vapour sorption behaviour of different types of hair samples were investigated by means of a DVS. Figure 5 shows only small differences between moisture uptakes in different hair samples at low humidity conditions, but at above 60% RH the moisture uptake of bleached hair fibres was significantly greater than the undamaged caucasian virgin and thermally treated hair fibres, which is in good agreement with the SEA wettability profiles shown in Figure 4.

From Figure 6 thermally treated hair fibres showed a much higher level of sorption/desorption hysteresis at >60% RH compared to the bleached or undamaged fibres, indicating that moisture diffusion out of the bulk is more restricted for the thermally treated sample. Also, water may penetrate deeper into the thermally damaged hair fibers.

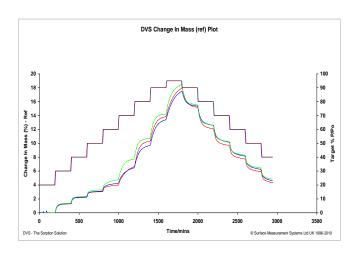


Figure 5. The superimposed moisture sorption and desorption kinetic results for hair samples; undamaged caucasian virgin (red), bleached caucasian / thin (green) and thermally treated (blue) at 25°C.

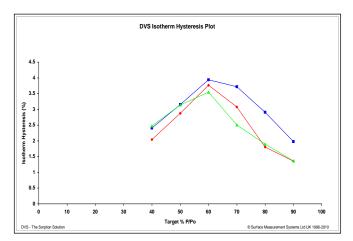


Figure 6. The superimposed DVS isotherm Hysteresis plots for samples undamaged (red), bleached (green) and thermally treated (blue) at 25°C.



Conclusion

iGC SEA and DVS techniques were used to determine the surface energetic heterogeneity and water sorption performance of undamaged caucasian virgin as well as thermally and bleached damaged hair samples. Undamaged hair sample was energetically heterogeneous with a relatively lower mean dispersive surface energy and lower moisture uptake level than the bleached sample, which showed higher mean dispersive surface energy value and wettability due to an increase in the number of high energy sites on the surface of hair.

References

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[3]Dong, S., Brendlé, M. and Donnet, J.B. (1989) Study of solid surface polarity by inverse gas chromatography at infinite dilution. Chromatographia, 28(9-10), p469-472

[4]Della Volpe, C. and Siboni, S. (1997) Some reflections on acid-base solid surface free energy theories. J. Colloid Interface Sci. 195, p121-136.

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^[1]Unbach. L. (editor), Cosmetics, Thieme, Stuttgart 1988