Measurement of water holding capacity using a condenser-chamber TEWL instrument

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Synopsis

The aim of this work was to develop a method for measuring equilibrium water uptake of small samples when exposed to different conditions of relative humidity (RH). Samples of excised human stratum corneum (SC), snake skin sheddings, nail clippings and hair were exposed to air with controlled RH of 22%, 33%, 43%, 53%, 75% ,81% and 95% for more than 48 hours prior to measuring desorption flux in the low humidity of a condenser-chamber TEWL instrument.

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Introduction

The measurement and control of the moisture content of materials is important to many industries, as it affects its physical properties and stability in storage. Materials where the control of moisture is essential include foods, pharmaceuticals, paper and board, wood, textiles, building materials and packaging.

Conventional methods of measuring water uptake use a microbalance to measure weight loss or gain in the presence of air of controlled humidity [1-2]. Such methods are relatively slow and expensive, and care needs to be exercised to reduce pressure dependent buoyancy, convection and thermal gas flow effects [2]. A recently developed high-resolution gravimetric flowing gas or Dynamic Vapour Sorption (DVS) instrument can measure the uptake and desorption of water vapour and organic vapours with a resolution down to 0.05µg [3].

Water vapour desorption measurements can also be performed using a condenser-chamber TEWL instrument [4]. Such instruments use a condensing surface whose temperature is held below the freezing point of water to control the humidity within the measurement chamber independently of ambient humidity. With a condenser temperature of -7.3ºC, as used in the experiments reported here, the RH in a sealed measurement chamber at an ambient temperature of 22ºC is 11.3%. Small samples, pre-conditioned at higher or lower RH will therefore lose or gain water when placed inside such a chamber, until they reach equilibrium with the chamber RH. The dynamics of the equilibration process can be studied by measuring time-series curves of associated water vapour flux. The total quantity of water lost or gained can then be calculated from such time-integrated flux curves.

The relationship between the quantity of water desorbed by a sample and RH is characterised by a sorption isotherm, as illustrated schematically in Figure 1.
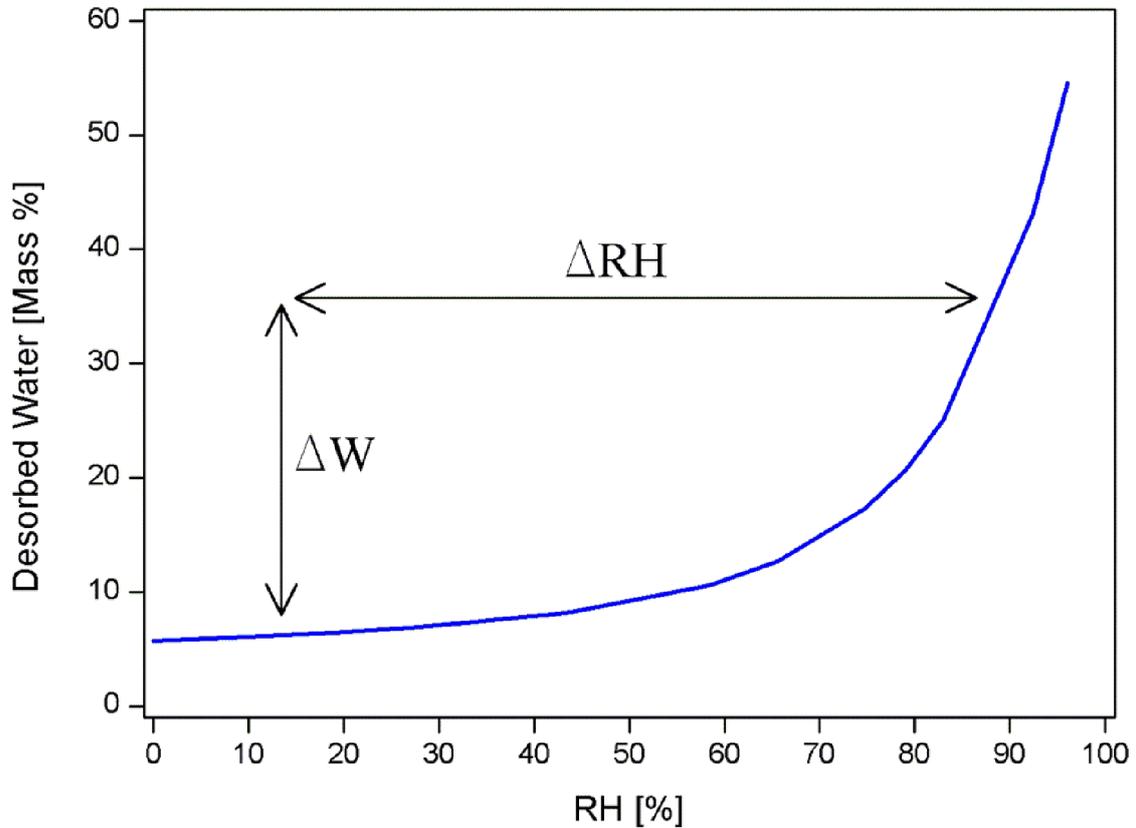

Fig 1: Schematic illustration of a sorption isotherm (curve) and its relationship with condenser-chamber measurements of desorbed quantity ΔW for a given change ΔRH from pre-conditioning RH to chamber RH.

Our measurements with the condenser-chamber method determine the change in the quantity of desorbed water ΔW for a change ΔRH from pre-conditioning RH to chamber RH. Therefore, measurements over a range of pre-conditioning RH values determine the form of the sorption isotherm relative to the equilibrium quantity of water in the sample at the RH of the chamber.

Experimental

Desorption experiments were performed using a prototype version of an AquaFlux AF100 condenser-chamber instrument (Biox Systems Ltd, UK), together with the apparatus shown in Figure 2.
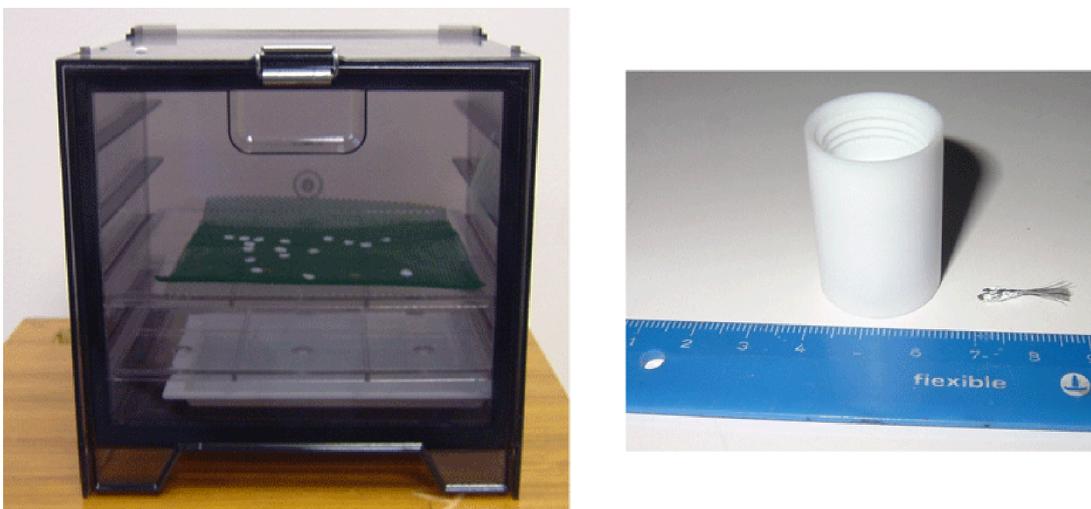

Fig 2: Apparatus for desorption measurement. Left: pre-conditioning chamber using saturated salt solutions for humidity control. Right: PTFE desorption cap for coupling onto the condenser-chamber measurement head, together with a sample of hair.

The measurements were performed at an ambient temperature of 22ºC and a condenser temperature of -7.3ºC. Samples of excised human SC, snake sheddings, hair and nail clippings were exposed to atmospheres of controlled humidity in the range 22% to 95% RH in sealed pre-conditioning chambers containing saturated salt solutions for at least 48 hours prior to measurement. Pre-conditioned samples were then placed into a PTFE desorption cap of 16mm well depth and screw-coupled to the AquaFlux measurement chamber. Water vapour flux density was then measured at 1.5 second intervals, until steady levels were reached.

Results and discussion

Figure 3 show examples of desorption curves for a range of samples, all pre-conditioned at 75% RH and measured at an ambient temperature of 22°C.
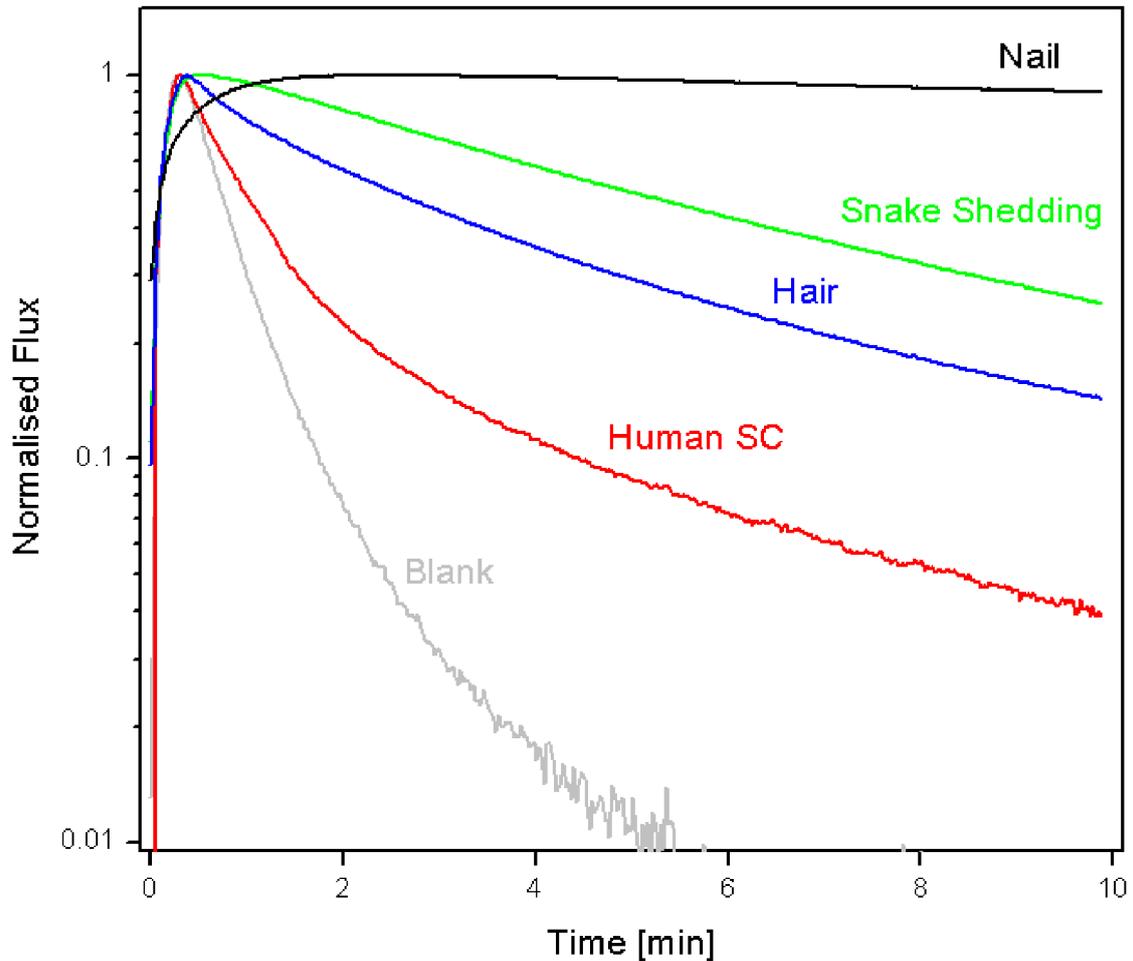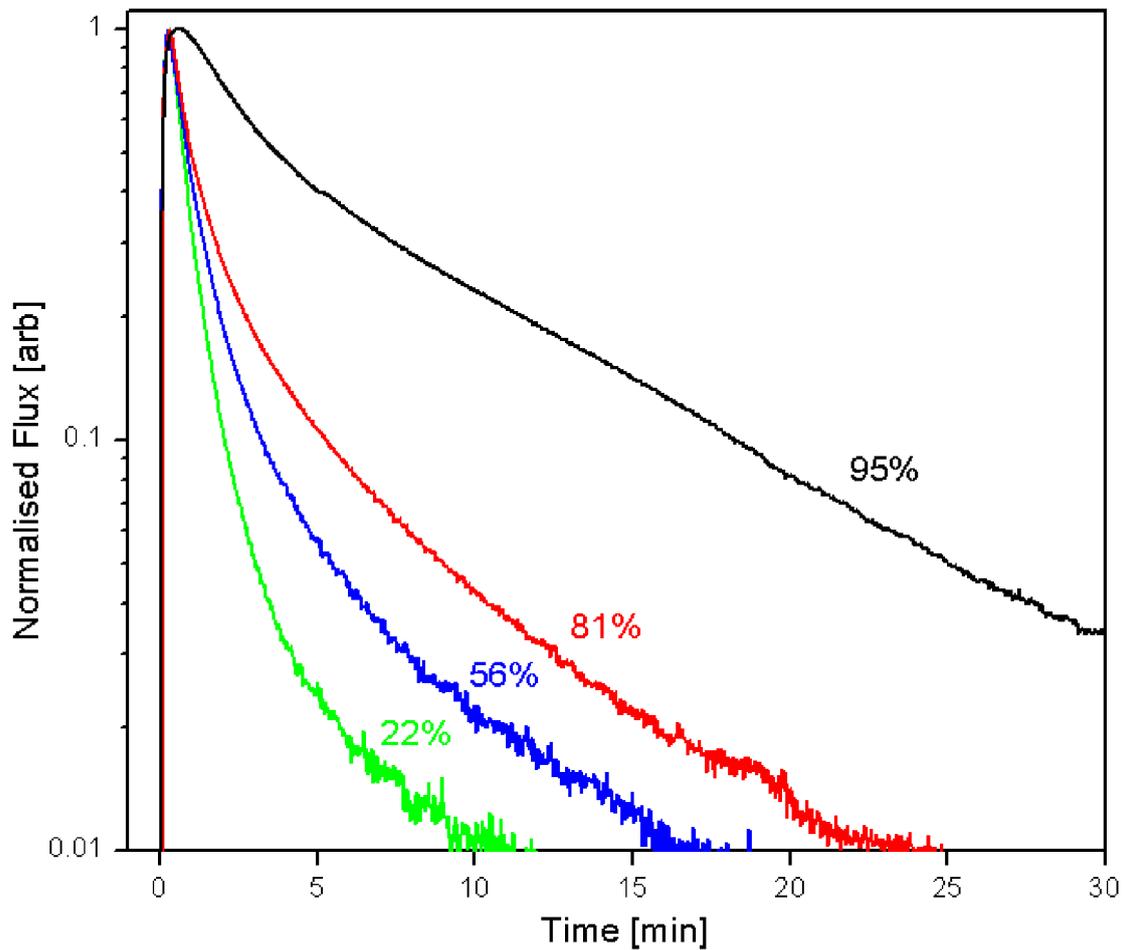

Fig 3: Desorption dynamics of a range of samples, all pre-conditioned at 75% RH.

A normalised, logarithmic flux scale is used to highlight the dynamics rather than the quantity of water desorbed. The blank curve was measured with an empty desorption cap, to indicate the instrumental response time. The desorption curve for excised human SC shows two distinct decay rates that may be associated with different water binding states. The curves for nail and snake shedding have rounded peaks that indicate the establishment of water diffusion

and associated concentration gradients within these materials. Nail has the slowest desorption rate, where 51 minutes are required in this case for the desorption flux to decay to half its peak value.

Figure 4 shows examples desorption curves for excised human SC samples pre-conditioned at a range of RH values.

Fig 4: Desorption dynamics of excised SC, pre-conditioned at the RH values shown.

As in Figure 3, a normalised, logarithmic flux scale is used to highlight the dynamics rather than the quantity of water desorbed. These desorption curves are clearly non-exponential, with fast initial decay rates that progressively decrease as the samples lose water. The distortion of the RH=95% curve is thought to be due to sample wrinkling during drying.

Desorbed quantities, expressed as a percentage of the weight of the sample, are presented in Figure 5 for excised human SC, snake sheddings, nail and hair.
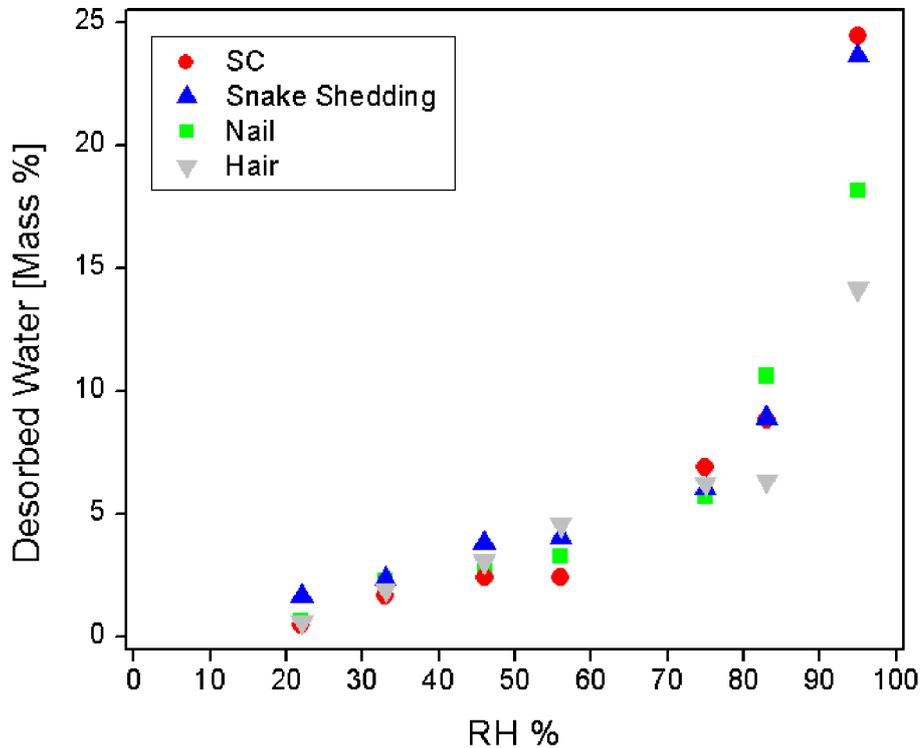
Fig 5: Desorption characteristics of various biomaterials. The relationship between these data and desorption isotherms is explained in Figure 1.

These were calculated by integrating the flux density time-series curves with respect to time. The data for excised human SC are in qualitative agreement with the results of Lévêque [5]. It is interesting to note that the desorption curves for excised human SC are similar to those of snake shedding, whereas nail and hair take up considerably less water at high humidity.

Conclusions

Desorption measurements with a condenser-chamber TEWL instrument give useful results quickly and economically. Work is now in progress to develop

mathematical models of the desorption dynamics in terms of water diffusion within the samples, from which (a) diffusion coefficients may be obtained and (b) measurement time may be reduced by extrapolation of early data.

Acknowledgements

References

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