Procedure for the Gravimetric determination of the Relative Humidity in standard-conditioned test laboratories

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Abstract

Samples must undergo physical tests, they require a prior conditioning in a controlled atmosphere for a time prior to the trials.
The main objective of this paper is to study the relationship between variations in the moisture content of a sample hygroscopic and moisture variations in a controlled environment and select the hygroscopic material more sensitive to variations in moisture in order to develop a new gravimetric method for measuring the relative humidity

Keywords: relative humidity, physical testing, drying.

1. Introduction

Leather samples for physical testing require prior conditioning in standard atmosphere for a period of time before being tested. According to the ISO 554-1976 Standard 23±2°C and 50±5% RH conditions are recommended, although alternative values of 20±2°C and 65±5% RH are admitted. The two possibilities are identified as 23/50 and 20/65 in the IUP 3 Standard.

Relative Humidity and Temperature standard conditions are achieved through forced circulation of air previously set at these conditions in a climatic unit. The contribution of heating resistance/cooling devices for temperature and that of steam-generating/drying (by cooling) devices for relative humidity control are needed.

Sometimes both heating and cooling actions should be needed in order to set into standard conditions a cooler and more humid external atmosphere, causing a situation of competition between heating and cooling actions of the climatic unit which results in specific situations where, occasionally, humidity and temperature exceed the tolerance limits. This normally occurs when sudden changes in external atmospheric conditions forces the climatic unit to balance the external conditions.

Although both humidity and temperature regulators are set at the standard conditions, it has been observed that these sudden variations in the external atmospheric conditions, sometimes induces deviations in relative humidity higher than 10% and, in temperature higher than 2ºC exceeding the tolerance limits, when measured by temperature and humidity sensors coupled to a data logger. Values of relative humidity recorded by the data logger differ from those of humidity yielded by the moisture content of calibrated leather samples gravimetrically measured.

The discrepancies between the relative humidity values yielded by the humidity sensor coupled to a data logger and those yielded gravimetrically by the calibrated hygroscopic samples can be attributed to the kinetic process of moisture absorption and desorption of the sample that is produced in a delay with the variations in the external moisture conditions (1). A sudden change in the external moisture out of tolerance limits induces a gradual variation in moisture content of the sample to reach the equilibrium. Although the external variations exceed the tolerance limits, moisture sorption and desorption is normally balanced along the time, resulting in variations of moisture content of the samples within the tolerance limits. The accuracy on the determination of the relative humidity based on the gravimetric determination of the moisture content of hygroscopic samples is influenced by the hysteresis phenomenon (2).

The main aim of the work is to study the relationship between the moisture content of the sample and variations in humidity of testing lab, to select the most sensitive hygroscopic material that allows us to develop a new method of gravimetric determination of relative humidity.

2. Materials and methods

Variations in moisture content of hygroscopic materials like cotton, hemp, cellulose (lints for paper), wool and leather gravimetrically measured, were related with the environmental variations in relative humidity of a non-controlled atmosphere along different periods of time. Variations in both the environmental relative humidity and the moisture content of the samples enabled us to select the hygroscopic material with the highest variations in sorption and desorption when compared with the observed variations in the environmental conditions. Wool, leather and leather treated with surfactants were tested in sorption desorption tests from 30 to 70% RH. The hysteresis in moisture content of samples has been determined by measuring the absorption and desorption of moisture in wool, leather and leather treated with anionic surfactant using the sorption analyzer DVS Q5000 form TA Instruments. Sample weights were between 20 and 25 mg.

Measuring procedure:

1) Samples conditioned in standard atmosphere 23/50 are pre-stabilized in the DVS chamber: temperature 23ºC, relative humidity 50%. When variation in weight is lower that 0.02% for 10 min, the step is concluded.

2) Sorption step: the sample previously stabilized at 50% RH is subjected to sorption tests at 70% RH. When variation in weight is lower that 0.02% for 10 min, the step is concluded.

3) Desorption step: the sample stabilized at 70% RH is subjected to desorption at 30% RH. When variation in weight is lower that 0.02% for 10 min, the step is concluded.

4) Sorption steps: the sample is subjected to sorption tests that progressively increase from 30% to 70% in steps of 10%RH. When variation in weight is lower that 0.02% for 10 min, the step is concluded.

5) Desorption steps: the sample previously stabilised at 70% RH is subjected to desorption steps from 70 to 30% in steps of 10%.

3. Results and discussion

The gravimetric determination of moisture content in % of different samples according to the variations in relative humidity measured by a RH sensor connected to a data logger showed that wool (2) and leather (3) were the samples with the highest rate of variation in moisture content per unit of relative humidity. Variations in wool and leather moisture content were respectively two and three times greater than variations in cellulosic samples.

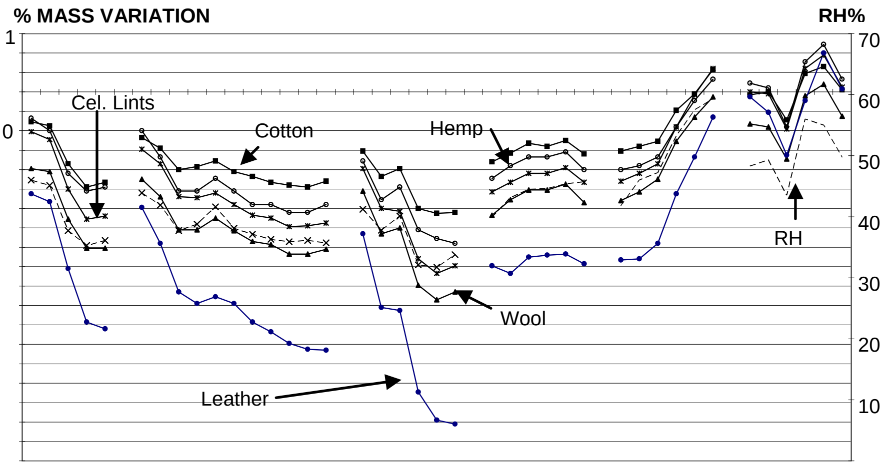

Figure 1: Mass variation in % db of different hygroscopic materials (cellulose lints – paper, cotton, hemp, wool and leather according to the variations in relative humidity of non-controlled climatic environment along different measuring periods of increase and/or decrease in relative humidity

The reduction of hysteresis phenomenon when samples are subjected to cyclic variations of relative humidities between 30 and 70% have been studied in order to find the most suitable materials to be used as sensors for the new gravimetric procedure of relative humidity determination. Authors have studied the influence of the application of surfactants on hide powder in the reduction of the hysteresis (2). Figure 2 illustrates this effect when moisture sorption and desorption tests were performed on two different hide powder samples between 0 and 100% of relative humidity (activity of water aw between 0 and1).

The variations in moisture content of wool, leather and leather treated with a surfactant measured at the end of each sorption/desorption step between 30 and 70% of relative humidity in steps 4 and 5 of the measuring procedure, were used to calculate the moisture content at the equilibrium fitting the parallel exponential kinetics PEK model developed by R. Kohler et al. (4). The methodology used to fit the non-linear regression PEK model was that explained by Manich et al (1) and the moisture contents at equilibrium at the end of each step are those presented in Table 1.

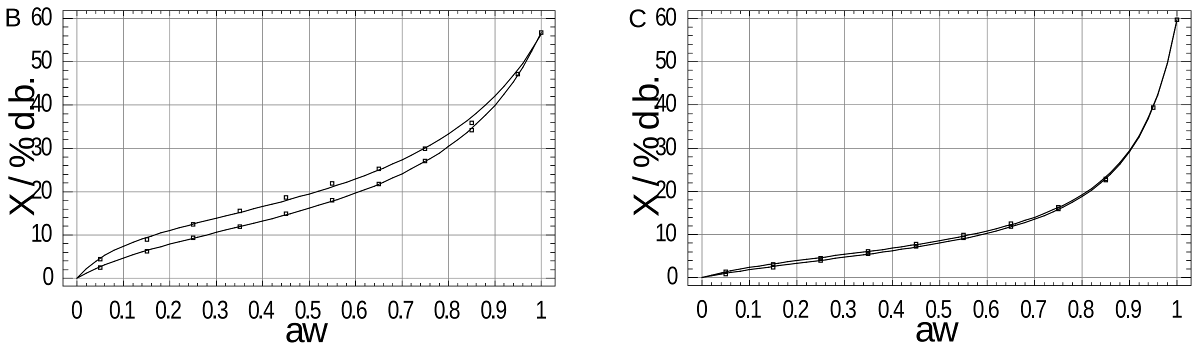

Figure 2: Variation in moisture content Moisture content of hide lyophilised powder (B) and surfactant treated hide powder (C) according to the relative humidity measured as water activity.

Table 1: Moisture content of wool, leather and surfactant treated leather (% db) at equilibrium according to the relative humidity in sorption and desorption tests.

Sample	Sorption steps from 30 to 70% RH					Desorption steps from 70 to 30% RH				
	Initial content	RH 40%	RH 50%	RH 60%	RH 70%	Initial content	RH 60%	RH 50%	RH 40%	RH 30%
Wool	8.05	9.03	10.01	10.98	12.23	12.18	11.32	10.40	9.34	8.02
Leather	12.85	14.04	15.02	15.93	17.30	17.21	16.17	15.18	14.16	12.71
ST Leather	13.53	14.33	15.00	15.66	16.40	16.35	15.75	15.13	14.43	13.49

Mathematical modelling

Regression analysis between moisture content (% db) of the samples and environmental relative humidity has been performed. The model that best fit moisture content to relative humidity showing the highest determination coefficients is the exponential one:

RH(%) = EXP(a - b/X), being X the moisture content of the sample (% db)

Table 2 shows the coefficients a and b of the predictive equations of relative humidity RH(%) in function of the moisture content of the samples X (% db), the determination coefficient R^2 (the square of the correlation coefficient expressed as percentage) and the standard error of the estimation (the square root of the residual mean square) which corresponds to the estimated standard deviation of the RH that is not explained by the model. Besides the experimental error of the procedure, this includes the effect of hysteresis on the variability of the estimation. The confidence intervals of the prediction at 50% of relative humidity with 95% of probability are also included.

Figure 3 plots the predictive regression equations including the 95% confidence interval of the prediction according to the different samples used. It is relevant that the more accurate prediction of the relative humidity are those given by leather samples and between these that given by the surfactant treated leather sample in spite of the shorter range of moisture content variation. Based on the confidence intervals at 95% of probability, it can be seen that the relative humidity of an environment can be predicted using calibrated samples of wool and leather with accuracy higher than ±1%. The best results were those given by the surfactant treated leather sample probably due to the decrease of hysteresis induced by the surfactant.


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Table 2: ***a*** and ***b*** regression coefficients of the predictive model of RH(%) in function of the moisture content of the sample X (% db), determination coefficient R^2 and Standard error of the estimation E and 95% confidence interval CI 95 at 50%RH.					
Sample	***a***	***b***	R^2	E	CI95
Wool	5.87	19.91	99.11	0.032	±0.097
Leather	6.72	42.49	99.53	0.023	±0.070
Leather surfactant treated	8.28	65.94	99.74	0.017	±0.052

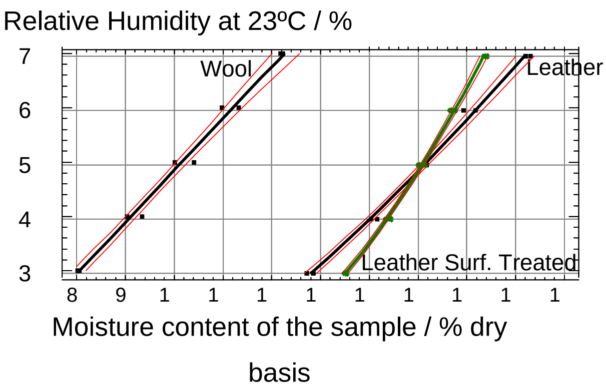

Figure 3: Prediction of the Relative Humidity in function of the moisture content of the samples in % d.b. including 95% of confidence intervals.

4. Conclusions

Hygroscopic materials, and in particular leather, are specially sensitive to variations in relative humidity. This makes them suitable for the development of a new method for the gravimetric determination of the relative humidity of standard lab.
The determination of the environmental humidity through direct measurement of the moisture content on calibrated leather samples enables us to get a more accurate estimation of the humidity conditions of the samples to be tested. This avoids the bias effect that can be attributed to an indirect determination of the humidity using a standard environment humidity sensor.

The accuracy of the proposed method in the prediction of the relative humidity can be higher than ±1% when used in environmental controlled laboratories. Optimization of the leather surfactant treatments are being considered in order to minimise the phenomenon of hysteresis.

5. References

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