Formaldehyde Release from Chekiang Lambskin, Characteristic Parameter Determination and Influencing Factors

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Abstract

VOC emissions may cause poor indoor (vehicle cabins included) air quality (IAQ). Recent studies have been conducted on the characteristics of volatile organic compounds (VOCs) emissions, most of them are focused on wood-based building materials, fur or fur imitations are scarcely mentioned. However, formaldehyde or formaldehyde-containing chemicals are often used in the production of fur, resulting in a certain amount of formaldehyde remaining in the finished product. This paper takes formaldehyde (FA) as an example to study its diffusion in fur. In order to understand and control the behaviour of the formaldehyde emission characteristics. Based on detailed mass transfer analysis of the emission process in a ventilated chamber, this paper measured the three emission characteristic parameters, *i.e.,* the initial emittable concentration (C_0), the diffusion coefficient (D_m) and the partition coefficient (K). The influence of different factors on the release parameters was also studied, as well as the time required for the emission of formaldehyde under different ventilation rates; we also compared the formaldehyde results of different test methods. The results showed that both D_m and C_0 increased as temperature rose, but C_0 increase is significant. With the increase of relative humidity, D_m decreased but C_0 increased. In addition, the prediction model agrees well with the experimental data. The greater the ventilation rate, the faster the improvement of air quality standards in the cabin; The initial concentration was less than 43% of the formaldehyde content obtained through the extraction method and less than 4.6% of the formaldehyde content obtained by the distillation method.

INTRODUCTION

Nowadays, individuals spend more than 80% of their time in an indoor environment such as staying at home, working in offices and public buildings. However, indoor air pollutants are several hundred times higher than those outdoors.[1,2] Volatile organic compounds (VOC), formaldehyde and ozone emitted from indoor building materials and furniture have become one of the major causes of degrading indoor air quality, which can negatively affect human comfort, health and productivity, which may cause general symptoms, such as headache; eye, nose, or throat irritations; dry cough; dizziness and nausea; difficulty in concentrating; tiredness. It is so serious that formaldehyde has been have classified as human carcinogen in European countries.[3,4]

However, the literature shows that studies on the effect of formaldehyde emissions mainly focus on interior decoration materials. Its effect on fur products is hardly available. Fur is widely used in products such as pillows, garments, carpets, metope adornments [such as living room wall decoration, wallpaper, TV setting wall, decorative board], and car mats *etc*. But in fur processing, the use of micro-biocides, tanning agents and straight hair fixative will lead to a certain amount of formaldehyde remaining in finished products. The toxic chemicals in the products may be released and transferred, doing harm to the environment and humans.[5-8] Therefore, it is of great importance to study the formaldehyde emission from fur.

The common methods used to describe, simulate and predict the formaldehyde emission process operates *via* two mathematical models: empirical and physical models. The former is based solely on statistical analysis of emission data obtained from environment chamber testing. Typical examples are the first-order decay model and the power-law decay model.[6] Although empirical models are simple and easy to use, they are not able to provide an insight into the physical emission mechanisms and can not be easily scaled from the test conditions to other conditions. The latter is based on mass transfer theory and follows the Fick's second law in which internal diffusion in the material is caused by the concentration, pressure or temperature gradient. The harmful material being distributed on an air-material interface is caused by diffusion and convective mass transfer. Since a physical model can reveal the law of material distribution, it is considered as the primary research method for modelling formaldehyde emission.

In a physical model, there are three key emission characteristic parameters: the initial emittable concentration C_0, the diffusion coefficient Dm and the partition coefficient K. The initial emittable

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concentration C_0 refers to the free emission of the formaldehyde concentration in the material. The diffusion coefficient D_m is the physical quantity which describes the formaldehyde diffusion speed. The partition coefficient K is the ratio of formaldehyde concentration to solid-phase and gas-phase at the material and air boundary layer.[9-13] Thus, the determination of the three parameters are essential to a formaldehyde emission study. The characteristic parameters are determined by using the environmental cabin [enclosure], monitoring the formaldehyde distribution process and equilibrium state of formaldehyde concentration changes, and a mathematical model to solve the solution.

The methods can be divided into two categories:

(1) Using the emission process to fit the parameters. Little *et al.*[14,15] used the mass transfer model to fit the experimental data to get three parameters; Xiong *et al.*[16] proposed that D_m and K were calculated by linear fitting of mass concentration footprint, and then C0 was deduced.

(2) Using an equilibrium state, such as multiple regression regression method,[17] variable gas ratio method,[18,19] multiple equilibrium regression method[20] and so on, through the experimental data fitting obtained, C_0 and K, and D_m need to be measured by mercury intrusion or other methods. In this paper, we adopted the ventilated chamber C-history method proposed by Yang Tao[21] for simultaneous determination of C_0 and D_m. The method is based on the release process of the VOCs in the ventilated chamber, the K value is preset, and then the logarithm of the formaldehyde concentration in the environment chamber and the corresponding time are linearly fitted. Finally, key parameters C_0 and D_m were obtained by slope and intercept. In addition to the determination of the characteristic parameters, the environmental factors will affect the release parameters, such as temperature, relative humidity and other environmental factors. Therefore, the impact of environmental factors on the release of parameters is also very important.

In this study, the formaldehyde emission experiment for fur was carried out in a ventilated chamber by changing temperature, relative humidity and ventilation rate respectively. The formaldehyde concentration in the environment was detected by the US Interscan4160-2 formaldehyde analyser. Based on the measured experimental data and with the help of the release model to solve the characteristic parameters, different factors influencing the characteristic parameters were obtained. Compared with D_m in the literature, the accuracy of D_m is verified, and the content of formaldehyde measured by C_0 in other test methods is obtained.

EXPERIMENTAL PROCEDURE

Materials

The fur samples used in the experiment are from the same batch of Chekiang lambskin which are available on the market. (Hereinafter referred to as the fur). Before use, the samples were trimmed into a uniform size and placed in a constant temperature and humidity chamber (temperature: 23°C, relative humidity: 50%), hung for three days, then sealed with a bag and stored in the refrigerator.

Equipment and instruments

A 1m^3 formaldehyde environmental chamber (QWH – 1000B) was obtained from Jinan Hainer Technology Co. Ltd. 4160-2, a formaldehyde analyser (0~19.9ppm, accuracy: 0.01) was purchased from the American Interscan Company.

Formaldehyde emission test of fur

The chamber was kept running under airtight condition with the required temperature, relative humidity and air change rate. When the formaldehyde content in the empty chamber became less than 0.08 ppm (background concentration), the fur samples were immediately placed into the chamber. The sampling interval (2min) was repeated until the step change between the formaldehyde concentration in the cabin was less than 5%, checked for three times, indicating that the release had reached a balance and the test was finished. The experimental scheme is shown in Table I.

TABLE I
Experiment scheme

Experimental group	Temp. (°C)	Relative humidity (%)	Air change rate (L/h)
1	18	50	1000
2	23	50	1000
3	30	50	1000
4	35	50	1000
5	23	40	1000
6	23	60	1000
7	23	50	10
8	23	50	500

Extraction method

The formaldehyde content in the fur products was tested according to ISO 17226-2.[22]

Distillation method

The samples were cut into particles, less than 4mm long and wide and evenly mixed. 1g of the mixed particles (accurate to 0.1mg) was placed in a glass still (500 mL) and distilled with 10% phosphoric acid (volume 1:9) 10mL, several glass beads and 100mL water under moderate heating.

The lower mouth of the condenser was inserted into a conical flask with 10mL distilled water added in advance (conical flask was placed in a mixed bath of ice and water to prevent the distillate from overheating and formaldehyde evaporating). Nearly 100mL was collected and transferred to a 100mL volumetric flask. The formaldehyde content in water was determined using a colorimetric method while a blank distillation was made.

Mass release model

Since individuals often air/ventilate rooms by keeping the windows and doors open, the experiment was carried on in a ventilated cabin rather than in a closed cabin. According to the model of formaldehyde emission in the literature[21] the simulation equation of formaldehyde over time in the ventilation cabin can be simplified as equation (1), and the parameters are detailed in paper 21:

$$C_a(t) = 2C_0\beta \frac{q_1 \sin q_1}{G_1} e^{-D_m\delta^{-2}q_1^{\,2}t} \quad (1)$$

Taking the logarithm of both sides of Equation (1) and simplifying it gives equation (2)

The purpose of changing (1) into equation 2 is to draw the relationship between $\ln C_{(a)}$ and time with $\ln C_{(a)}$ as the ordinate and time(t) as the abscissa. Then the slope and intercept were obtained from the relationship diagram, and the release parameters were obtained.

$$\ln C_a(t) = -D_m\delta^{-2}q_1^{\,2}t + \ln(2C_0\beta \frac{q_1 \sin q_1}{G_1}) \quad (2)$$

So, in the ventilation cabin, as long as the formaldehyde concentration and the relationship with processing time obeyed (2) as shown in the formula, the slope and intercept can be obtained by linear curve fitting. After that, by presetting *K* value (*K* value for the release of the influence of the concentration of parameters and hold discussion in sensitivity analysis) and using software 1 stopt (See Appendix), then D_m and C_0 can be determined directly.

RESULTS AND DISCUSSION

Effects of different temperatures on the release parameters

The formaldehyde release experiment for fur products was carried out by means of the appropriate test protocol. The formaldehyde concentration at different temperatures over time was thus obtained. For the accuracy of the fitting results, eight data points distributed in the middle and late stages were selected as the fitting of equation (2). The fitting results are shown in Fig. 1, where R^2 is greater than 0.9, showing the test results are as expected.

The straight line slope and intercept were obtained based on the fitting. D_m and C_0 were obtained by using the software 1stopt solution where *K* is 5000. As is shown in Table II, when the temperature was increased from 18 to 35°C, the diffusion coefficient D_m increased by 87%, and the initial concentration C_0 increased by 128%, respectively, which was mainly attributed to the temperature rise leading to the average kinetic energy increase of the residual free and adsorbed formaldehyde molecules in the fur. The molecular thermal motion increase resulted in increased diffusion intensity and diffusion extent. As a result, D_m and C_0 increased[23] which is consistent with the research results of Xiong Jianyin[24] and Yang Tao.[21]

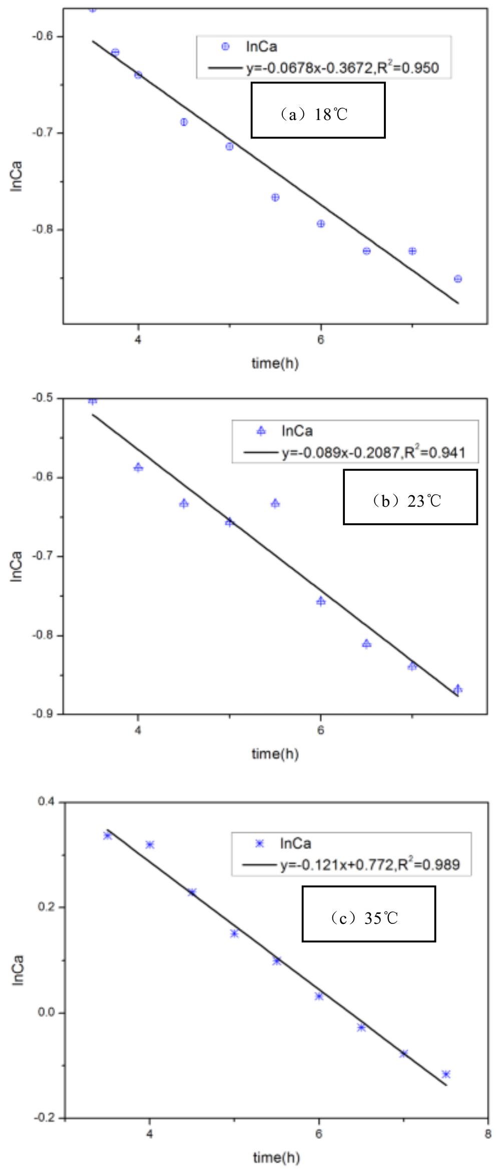


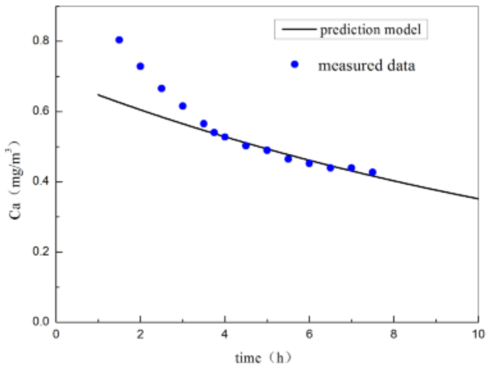
Figure 1. Linear fitting results of formaldehyde emission.

TABLE II
Characteristic parameters of formaldehyde emission

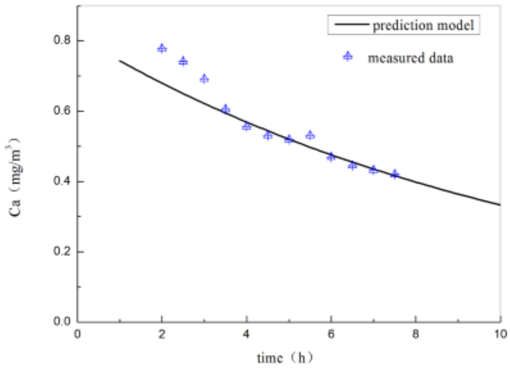
Temperature (°C)	D_m(m²/s)	C_0(mg/m³)
18	4.485×10^{-12}	1.746×10^{5}
23	5.934×10^{-12}	2.025×10^{5}
30	8.242×10^{-12}	2.426×10^{5}
35	8.373×10^{-12}	3.981×10^{5}

The diffusion coefficient D_m is the physical quantity which describes the formaldehyde diffusion speed, the initial emittable concentration C_0.

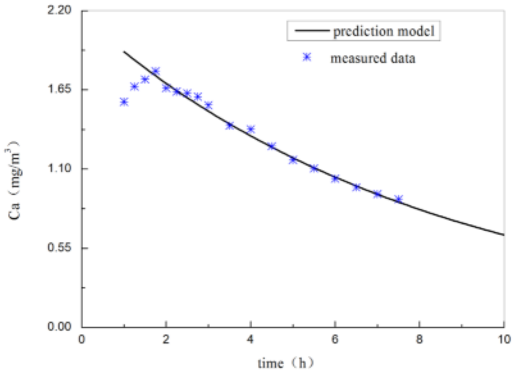
The characteristic parameters are brought into equation (1) to obtain the prediction model. As shown in Fig. 2, most of the actual measurement data falls on the curve of the prediction model. The deviation only appeared before the formaldehyde release. The actual measurement error was lower than 20%, so the characteristic parameters are considered to be accurate.[25]



(**a**) 18°C



(**b**) 23°C



(**c**) 35°C

Figure 2. Comparison of model predictive value and experimental values.

The effect of different relative humidity levels on characteristic parameters

The linearity of formaldehyde emission for different humidity levels is shown in Fig. 3, where R^2 is greater than 0.9, showing that the fitting result is better.

The characteristic parameters D_m and C_0 are shown in Table III. As the relative humidity increased from 40% to 60%, the diffusion coefficient D_m decreased by 22.34%, the initial concentration C_0 increased by 81.93%, indicating that the relative humidity for the C_0 is greater than that of D_m.

TABLE III
The *Dm* and *C*0 of formaldehyde release at different humidity levels

Relative humidity (%)	D_m(m^2/s)	C_0(mg/m^3)
40	9.412×10^{-12}	5.865×10^{4}
50	7.477×10^{-12}	8.511×10^{4}
60	7.309×10^{-12}	1.067×10^{5}

The diffusion coefficient D_m is the physical quantity which describes the formaldehyde diffusion speed, the initial emittable concentration C_0.

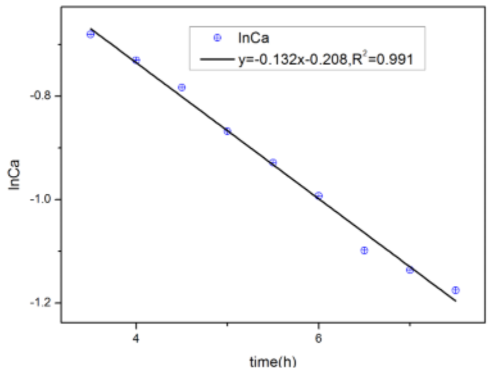
The primary reason for the decrease of D_m is that the diffusion intensity was weakened with the increase of relative humidity, the decrease of air density and the density difference in the diffusion direction becoming smaller.[26] The increase of relative humidity and C_0 is due to the formation of formaldehyde[27] which led to the reduction of air side formaldehyde content. As a result, the free formaldehyde in the fur is more likely to diffuse. Thus the initial concentration increased with the increase of relative humidity. The result is consistent with the report of Meining[28] and Zhang Jun.[29]

The comparison between the prediction model and the actual measurement data is shown in Fig. 4. Most of the experimental measurements are also on the prediction model.

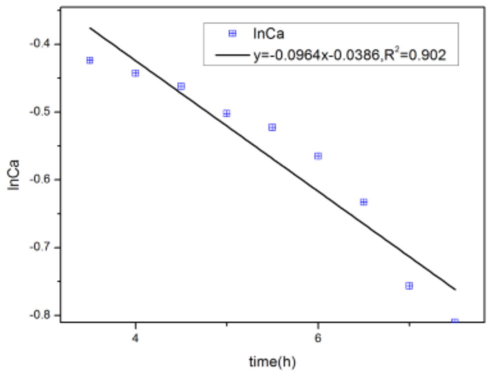
Comparison of diffusion coefficient D_m between fur and other materials in the literature

The diffusion coefficient D_m characterises the strength of the formaldehyde diffusion process for fur. The key parameters for the determination of formaldehyde in the literature[27] and in[31] are shown in Table IV.

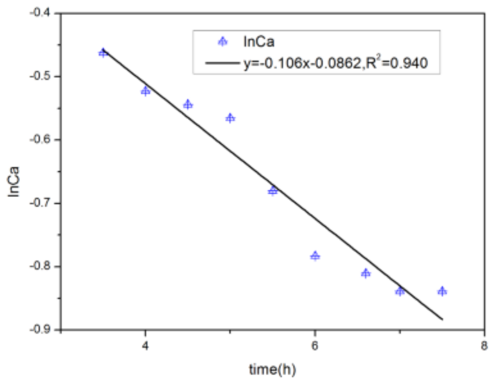
It can be seen from Table IV that the diffusion coefficient D_m of man-made panels, floor leather and carpet is generally in the order of 10^{-13} to 10^{-11}, and the formaldehyde diffusion coefficient of fur in this paper is 10^{-12} which is within the same order as that of carpet. It is probably because, fur, being a kind of carpet, the order of magnitude of its diffusion coefficient is the same as that of carpet. It is also due to the diffusion coefficient $D=Dr\varepsilon/\tau$ where: Dr Aperture is the material reference diffusion coefficient, ε is the porosity, τ is the sinuosity. Therefore, when the diffusion material is equilibrated with the external conditions, the diffusion coefficient is determined by the type of the diffusion material; as plate structure differs with the type, material, adhesive and the production process, the pore structure is not the same. In general, the processing of the sheet is greater than that of the carpet, resulting in a more dense pore structure than that of the carpet; τ is used to correct the distance increased in the diffusion direction because of the path twists and turns, generally it is a number greater than 1, for a porous medium, the value is 1.5~2.0, with compaction of the porous medium, its value can be as high as 7~8.[30] The floor structure is significantly denser than that of the carpet and fur, therefore, the formaldehyde molecules in the floor have to go through a longer path in the release process.



(**a**) 40%



(**b**) 50%



(**c**) 60%

Figure 3. Linear fitting results of formaldehyde emission.

Comparison of release time of formaldehyde in the environmental cabin under different ventilation conditions

In the $1m^3$ environmental cabin, whilst other experimental conditions were kept unchanged, the air change rate was set to 10L/h, 500L/h, 1000L/h, respectively.

Table V summarises the equation of the formaldehyde concentration change with time in the environmental cabin. It was obtained according to the experimental data of formaldehyde change with time and the method described in Chinese National Standards to fit the data in combination with Equation 1. Based on the Chinese national standard[31] of the indoor air quality standards for formaldehyde content ($0.1mg/m^3$), the time required for reducing formaldehyde concentration in the environment to indoor air quality standards was calculated . The results are shown in Table V.

It can be seen from Table V that when the ventilation rate is 1000L/h, the formaldehyde concentration in the cabin is 24.9h the formaldehyde content is less than half of that at the ventilation rate of 10L/h. Thus, with the increase of ventilation rate, the formaldehyde content in the environmental compartment was lowered to the standard, revealing that the ventilation rate has a positive effect on formaldehyde emission. This is because when the ventilation rate is high, more formaldehyde-free air came into the cabin. As a result, formaldehyde in the cabin was diluted. What's more, some formaldehyde was taken out of the cabin.[32]

Comparison of the initial concentration of fur formaldehyde with the concentration measured by distillation and extraction method

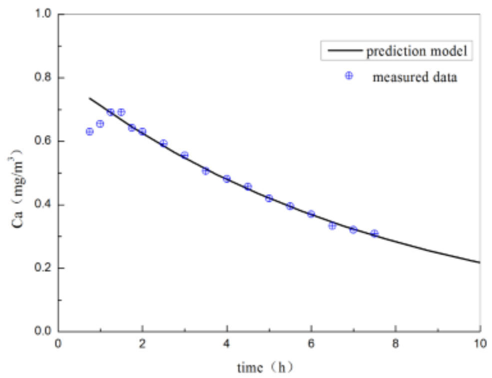
The initial concentration C_0 of formaldehyde in the fur was compared with that of the formaldehyde content as extracted by the extraction method and the distillation method at different temperatures. The initial sporadic concentration is defined as the total amount of formaldehyde that the fur can release when the formaldehyde concentration in the air medium is zero. The results are shown in Table VI. The initial concentration of formaldehyde is not more than 43% compared with that measured by extraction and is only 4.6% compared with that measured by distillation. This is mainly because formaldehyde measured by the release method is a part of the free formaldehyde in the fur whilst, the formaldehyde measured by the extraction method is that free in fur plus that absorbed in capillaries and that which is partially reversibly bonded. In addition to the free and adsorbed formaldehyde, all the reversibly bound formaldehyde is released with acid under high temperature. Therefore the formaldehyde measured through the distillation method is the total formaldehyde content in the fur,[33,34] indicating that most of the formaldehyde in the fur product is not released at room temperature. Therefore, the formaldehyde detection method should be based on different detection and detection media.

Sensitivity analysis

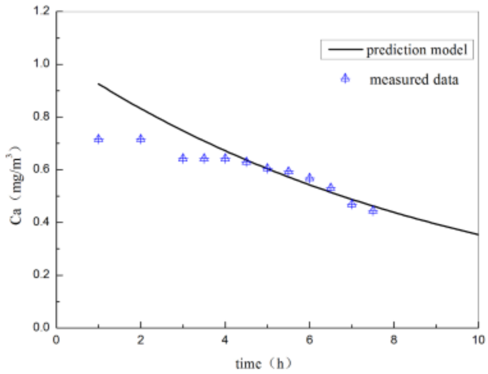
To solve D_m and C_0, a K value should first be set, so it is necessary to confirm whether the K value is reasonable or not. With a temperature of 23°C, relative humidity of 50%, ventilation rate of 1000L/h and the K values of 500, 1000, 3000, 5000, the corresponding D_m

TABLE IV
D_m values for different materials

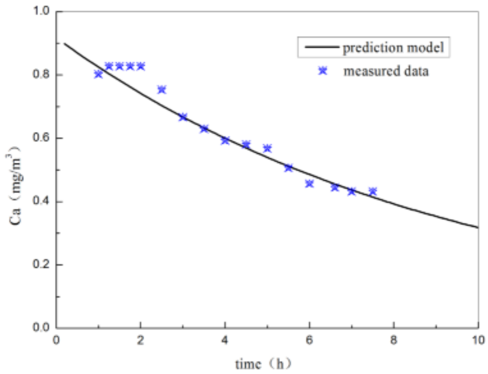
Materials	Fur	Plywood	Floor leather	Carpet	Composite board	Composite floor
D_m	5.934×10^{-12}	2.3×10^{-11}	5.2×10^{-13}	1.6×10^{-12}	2.3×10^{-13}	8.9×10^{-13}



(**a**) 40%



(**b**) 50%



(**c**) 60%

Figure 4. Relative humidity model predictive value and experimental value comparison chart.

TABLE V
Comparison of distribution time of formaldehyde in environmental chamber at different ventilation rates

Air change rate (L/h)	C_a(t) (mg/m³)	C_a(t) = 0.1mg/m³ time needed (h)
10	$1.29e^{-0.0369t}$	69.30
500	$1.33e^{-0.0976t}$	26.51
1000	$0.917e^{-0.089t}$	24.90

C_a(t) is time needed to reduce formaldehyde levels to the Chinese National Standard level. In hours.

and C_0 were calculated. As shown in Fig. 5, when the *K* value increased from 500 to 5000, D_m increased to 4.77%, C0 increased 2.02% ,the shift was less than 5%. So it can be determined that the change of *K* value has little effect on D_m and C_0.

TABLE VI
Comparison of the initial concentration of fur formaldehyde compared with that measured by distillation and extraction methods

Temp. of test (°C)	C_0 (mg/m³)	Formaldehyde measured by extraction (mg/m³)	Formaldehyde measured by distillation (mg/m³)
18	1.746×10^5		
23	2.025×10^5	9.268×10^5	8.734×10^6
30	2.426×10^5		
35	3.981×10^5		

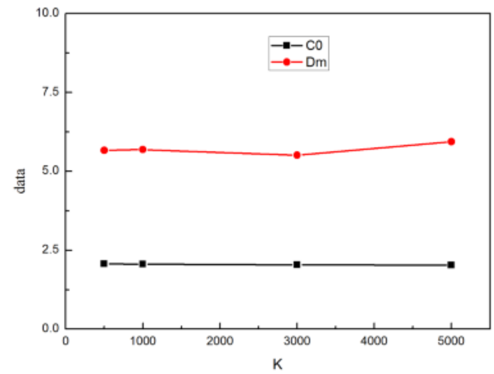
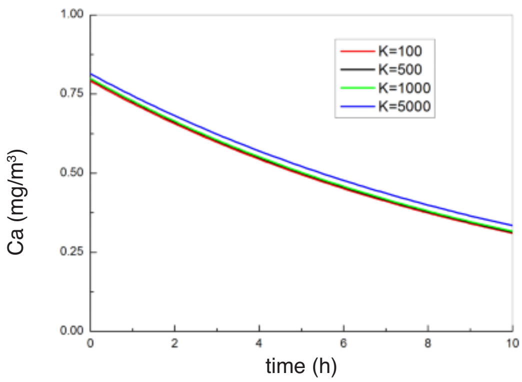


Figure 5. D_m and C_0 curves with *K* change.

Since K, D_m and C_0 are used to solve C_a, the change in *K* value also has a corresponding effect on C_a, so we also studied the effect of *K* on C_a. Different C_a values



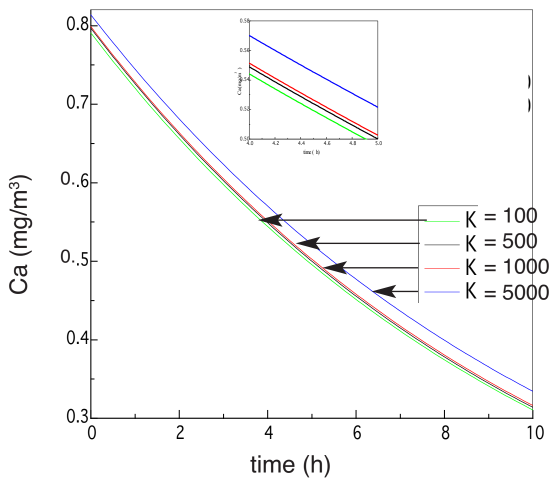


Figure VI. Changes of formaldehyde concentration in environment with K change.

were obtained by taking K = 100, 500, 1000, 5000. As shown in Fig. 6, the K value increased by 50 times (from 100 to 5000), but the change in C_a does not exceed 1.5%. Therefore, it can be decided that the value of K has little effect on the concentration of formaldehyde in the environmental chamber. Therefore, the above description using the default K value to solve the formaldehyde content in the environment chamber method proves to be reasonable.

CONCLUSIONS

The process of formaldehyde emission for fur products was studied using the formaldehyde emission model as used in the literature.

The conclusion was drawn as follows:

(1) the mass transfer model is applicable to formaldehyde distribution process in fur;

(2) As temperature rises, both the diffusion coefficient D_m and the initial concentration C_0 increase, and the increase of C_0 is greater than $_m$, but with the increase of relative humidity, D_m decreases while C_0 increases;

(3) The value of measured characteristic parameters D_m in this study is in the reasonable range as D_m values in the literature.

(4) The initial concentration of formaldehyde in the fur measured by the mass transfer model is less than 43% of the formaldehyde content by the extraction method and less than 4.6% of the formaldehyde content measured by the distillation method;

(5) By analysing the influence of K on D_m C_0 and C_a, it can be concluded that the value of K has a reasonable influence on the solution of the release parameter and the prediction model, so the preset K value can be used to solve the release parameter.

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APPENDIX

'1 stopt' software is the leading software platform for non-linear curve fitting in comprehensive optimisation analysis and calculation especially in the fields of non-linear regression, curve fitting, parameter estimation and solution of non-linear complex engineering model, etc. The advantage of the software is that the user does not need to give the initial value of the parameters, but gives it randomly by '1 stopt' software and finally finds the optimal solution through its unique global optimisation algorithm.

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