Model smoke stream adsorption over cellulose acetate stick with three-dimensional temperature gradient by combining in-situ DRIFTS with infrared thermal imaging

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Abstract Understanding the adsorption of the smoke stream (SR) on cigarette filter—cellulose acetate stick with different temperatures is beneficial for controlling chemical emissions and reducing the toxic effect of smoking on human health. However, the investigation of corresponding adsorption properties was missing because the adsorption of smoke stream substances on cigarette cellulose acetate is sensitive with the three-dimensional temperature gradient. In this work, the adsorption of typical smoke substances, such as CO, propylene glycol, formaldehyde, and acetone, on cellulose acetate stick were studied by in-situ diffuse reflectance Fourier transform infrared spectroscopy with different temperatures assisted by the infrared thermal imaging method. The adsorption capacities of cellulose acetate stick to these typical smoke substances is dependent on the adsorption time and temperature. The adsorption properties all fitted well with the Freundlich model. By a spectroscopic and mathematical explanation, quantifying contours of adsorption was performed. The 3D model of the normalized CO adsorption of cellulose acetate stick versus the spatial coordinates and time was established. This study gives unparalleled insight into smoke release characteristics of cigarette filtered by cellulose acetate and regulatory mechanism of cellulose acetate stick for reducing the negative effect of smoke on human health.

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Introduction

Cigarette smoke is a complex mixture produced by tobacco combustion, cracking and distillation (Boué et al. 2020). CO and low-carbon aldehydes and ketones are toxic components of cigarette smoke stream, seriously affecting human health (Chang et al. 2021; Starek and Podolak 2009). Over the years, a number of studies have investigated the harmful effect of tobacco and smoke on human health by measuring the combustion and pyrolysis characteristics and smoke toxicity of tobacco and testing the human biological characteristics (Adam et al. 2006; Heide et al. 2021). In particular, extensive studies have been undertaken to modify cigarette intercept processes to reduce harmful substances in smoke, in order to protect human health (Rostami and Hajaligol 2003). The release of CO and low-carbon aldehydes and ketones is an important index for measuring the combustion and pyrolysis characteristics and smoke toxicity of tobacco, as well as for reflecting the comprehensive quality of cigarettes (Li et al. 2002). It should be pointed out that the release of CO and low-carbon aldehydes and ketones over filters is greatly affected by the combustion and pyrolysis environment, especially by temperature (Pauwels et al. 2018). During puffing, air influx drives rapid changes in gas-phase and solid-phase temperatures (Baker 1974). Therefore, the investigation of their adsorption and retention characteristics at different temperatures is beneficial for guiding the design and development of filtration materials for cigarettes, and the optimization of cigarette filter, comprehensively improving the sensory quality and safety of cigarette smoke.

At present, the main cigarette filtration materials include cellulose acetate, polypropylene, and polyethylene terephthalate, etc. (Chau et al. 2020; Dong et al. 2021; Zhang et al. 2020). Among them, cellulose acetate stick as the cigarette filter can balance the smoke substances by trapping the excessive concentration of harmful cigarette smoke (Dwyer and Abel 2015). At the same time, it exerts a filtering role, intercepting smoke powder particles and adjusting suction resistance (Du et al. 2015). Previous studies investigated the overall transfer of CO, 1, 2-propanediol, glycerol, nicotine and some flavor components in different heated cigarette materials, aerosols and filters, and the change of the smoking cycle (puffing-smoldering-puffing) release of main components of smoke substances (Li et al. 2018; Baker 2006). Some
research groups have also studied the smoke substances release law of filter material and length on the main components of smoke substances under different heat sources, smoke core sections and suction conditions (Wen et al. 2014).

Therefore, understanding the adsorption of the smoke substances on cellulose acetate stick as cigarette filter with different temperatures is beneficial for controlling chemical emissions and reducing the toxic effect of smoking on human health. However, the change law of the adsorption structure and adsorption capacity of cigarette smoke component chemistry on the surface of cellulose acetate stick with temperature/coordinate axes is challenging, because the adsorption of CO and low-carbon aldehydes and ketones on the filter material is sensitive to the three-dimensional temperature gradient which is difficult to obtain on the filter stick.

In-situ infrared spectroscopy is useful non-invasive and real-time device for evaluating changes in a chemical structure and monitoring the changes of characteristic functional groups with temperature (Meunier 2016; Proaño et al. 2019; Shi et al. 2020; Zhong et al. 2020). Among the available characterization modes of infrared spectroscopy, diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) mode is the most suitable for in-situ characterization for gas–solid interface reaction, as it can collect and analyze the surface and interface information carried by the electromagnetic radiation reflected from the surface (Dzara et al. 2019; Li et al. 2020; Tofan-Lazar et al. 2013). Yet, for the measurement of the three-dimensional temperature gradient of the filter stick, the surface contact between the measuring instrument and the measured object can disturb the original temperature field of the object, affecting the transmission of the temperature sensing element, and leading to the inaccurate output signal (Li et al. 2014, 2016). Fortunately, the infrared thermal imaging technology does not interfere with the temperature of the measured object without inertial effect in the transmission of optical information, which can smoothly change the path and ensure the integrity of data (Jia et al. 2017; Pham Xuan et al. 2021; Roehl et al. 2009).

The dynamic changes in the three-dimensional temperature gradient and the adsorption characteristics of typical smoke substances are two important parameters to investigate the release of smoke for controlling chemical emissions and reducing the toxic effect of smoking on human health. In this study, we used infrared thermal imaging technology to detect the three-dimensional temperature distribution information of cellulose acetate stick of burning cigarette. Based on this, we used in-situ DRIFTS to characterize the adsorption of CO, propylene glycol, and typical low-carbon aldehydes and ketones, including formaldehyde and acetone, on the surface of cellulose acetate sticks at the above temperature range of the cellulose acetate stick. The corresponding adsorption kinetic model were established. This work may provide a new perspective for guiding the design and development of filter stick materials for cigarettes and improving the suction safety of filters.

**Experimental**

**Materials and reagents**

The chemicals used in the experiments, including formaldehyde solution, acetone, and propylene glycol were purchased from Sinopharm Chemical Reagent Co., Ltd. Deionized water (resistivity of 18.2 MΩ) was purified by using a Millipore system. Gases used in the study were Argon (Ar, 99.99 + %) and the compressed air (20 vol% O2/N2).

The cellulose acetate sticks used in this study were obtained from Eastman Shuangwei Fibers Company Limited. The degree of acetyl substitution of cellulose acetate is between 2.0 and 2.7. Its molecular formula is \[\text{[C}_6\text{H}_7\text{O}_2\text{(OCH}_3\text{)}\text{x(OH)}_3\text{-}], n = 200–400\]. It is widely used as cigarette filter tow (cigarette cellulose acetate tow) in commercial with a radius of 4 mm, and a length of 20 mm.

**Characterization of samples**

The morphology of the samples were investigated by a scanning electron microscope (Sirion 200). CO-TPD was performed on AutoChem II 2920. Approximately 80 mg of the sample was loaded into a U-type quartz tube, which was mounted on the instrument. Then the sample was pretreated in Ar at 100 °C for 0.5 h (heating rate 10 °C/min). After cooling to room temperature, 5% CO in Ar was passed through the sample for 1 h, with subsequent flushing with helium at 100 °C for 1 h. The TPD analysis was carried out in
flowing Ar from 50 °C to 300 °C at a heating rate of 10 °C/min.

Infrared thermal imaging test

The three-dimensional temperature gradient of the filter stick during cigarette burning was observed and recorded by the Forward Looking Infrared (FLIR) thermal imaging system with a THERMACAM 25 camera and the THERMACAM Reporter 2000 software. The distance between the camera and the object was fixed from 10 to 50 cm.

In-situ DRIFTS measurements

To elucidate adsorption on the surface of the CO and typical low-carbon aldehydes and ketones, in-situ DRIFTS measurements were performed on a Bruker IFS 66v/s FTIR spectrometer equipped with a self-built setup and a DRIFTS cell. As shown in Fig. 1, the setup consists of a detection system, a reaction system, and a coupling reaction gas-dosing system. In the gas-dosing system, mass flow controllers were used to control the 20 vol% O2/N2 compressed air which carried SR vapor from the saturator containing typical low-carbon aldehydes and ketones (LCA) or propylene glycol (PG), such as formaldehyde solution, acetone, and propylene glycol. The water vapor was supplied and regulated to the cell via a by-pass line. The relative humidity in the cell was determined using an electronic hygrometer fixed in the by-pass line. The reaction system consists of a praying mantis DRIFTS accessory (Harrick Scientific) and a reaction cell (HVC, Harrick Scientific). The reaction cell is equipped with a sample cup with retaining plates in it and covered by a dome fitted with three windows (Fig. S1). Cooling water was circulated through a coil surrounding the base of the dome to facilitate the reaction at room temperature.

Results and Discussion

Understanding the change of temperature field inside a burning cigarette is an important step in controlling the interception of smoke substances. The geometric model of cellulose acetate stick and its axial definition used in this study is shown in Fig. 2. A cellulose acetate stick has a radius of 4 mm, and a length of 20 mm, and uniform agglomeration properties of three-dimensional space structure and tow-bonding structure (Fig. 3).

The temperature field in the cellulose acetate stick was detected by infrared thermal imaging. The FLIR
camera based on the thermal imaging method was used to analyze the temperatures of cellulose acetate stick with cigarette burning. FLIR camera can detect minimum changes in temperature (0.1 degrees). The coordinate of the corresponding points in the 3D space can be obtained during temperature information acquisition according to the constructed 3D temperature model. Regarding temperature field simulation, the following conditions for cellulose acetate stick were declared: (1) the porous medium is even and isotropic; (2) the porosity, specific heat capacity, density, and heat transfer coefficient are constant; (3) natural convection and radiation are ignored (Jiang et al. 2018).

As shown in Fig. 4a, b, the temperature field distribution of the same cross-section was axially symmetrical. When the burning spot was almost close to the cellulose acetate stick (Fig. 4b), the highest temperature of the position in the cellulose acetate stick was 930°C which is observed closest to the burning tobacco. The temperature gradually decreased from the core temperature of 930°C to the edge temperature of 260°C along the r-axis. The heat diffused along the r-axis and x-axis, and the temperature distributions along the two axes were different. The core temperature from 930°C at x = 0 gradually decreased to 25°C at x = 16 mm approximately, and then remained stable.

From the infrared thermal images results, we obtained 3D temperature distribution information with the fitting curves in the r-axis and x-axis (Fig. 5a, b). The temperature distribution of the 2D cross-section under different x coordinates was different. The highest temperature was always in the central region. The temperature gradually decreased along the radial direction. Take the 2D cross-section at x = 10 mm for example (Fig. 5a), the curve of temperature versus r coordinate (Fig. 5b) can be fitted to a polynomial equation as follows.
Fig. 4  The infrared thermal images for a burning cigarette using cellulose acetate stick as a filter. a The 2D cross-section temperature distribution at \( x = 0 \) mm, the dotted circle represents the cross section of cellulose acetate stick; b the temperature distribution of a cigarette along \( x \) axis, the dotted cylinder represents the cellulose acetate stick; which were recorded when the burning spot is almost close the cellulose acetate stick

Fig. 5  a The radial temperature distribution of the cellulose acetate stick along with \( r \)-axis with b the fitting curve of radial temperature versus \( r \) at the cross-section of \( x = 10 \) mm; c The 2D cross-section average temperature distribution of the cellulose acetate stick along the \( x \)-axis with d the fitting curve of temperature versus \( x \); the insets are the fitted polynomial equations
\[ T = 15.21r^3 - 77.80r^2 + 88.11r + 647.9 \]  

(1)

The fitted equations of temperature distribution for all the 2D cross-sections under different \( x \) coordinates could be established in the same way as that at \( x = 10 \) mm. Based on this, the average temperatures of all the cross-sections of the cellulose acetate stick could be easily acquired by the temperature integration of the 2D cross-section. The highest average temperature was observed at \( x = 0 \), which is closest to the burning tobacco in the course of smoking; then the heat of 2D cross-section at \( x = 0 \) diffuses along the \( x \) axial-flow direction, and the temperature of 2D cross-section decreased to room temperature at \( x \approx 16 \) mm. The curve of the average temperature of 2D cross-section along the \( x \)-axis could also be calculated using a polynomial equation:

\[ T = 0.0588x^3 - 0.1603x^2 - 48.18x + 613.55 \]  

(2)

Which was shown in Fig. 5d. The R-squares of the two equations were close to 1.0, indicating the good predictability of the two fitted models. Additionally, the temperature gradients in the cellulose acetate stick were reduced both along the \( x \) axial and radial directions. Through the acquisition and mathematical model analysis of the typical temperature data of cellulose acetate stick, the 3D temperature distribution model could be obtained. According to the 3D temperature distribution model, the temperature field of the whole cellulose acetate stick could be observed. On the other hand, we could easily get the temperature information at any point in the cellulose acetate stick from the obtained 3D temperature distribution model.

In-situ DRIFTS study of the surface-adsorbed species is powerful for investigating the adsorption kinetics. After determining the 3D temperature distribution of the cellulose acetate stick mentioned above, in-situ DRIFTS technique was used to test the adsorption characteristic of typical smoke substances, such as CO, formaldehyde, acetone and propylene glycol, in the temperature range of 25–930 °C. Firstly, as one of the main harmful substances in the smoke stream, CO was used as a probe molecule to test the adsorption characteristic of cellulose acetate stick at different adsorption temperatures for 12 min. Figure 6 displays the variation of DRIFTS spectra of CO adsorption and the DRIFTS differential spectra of CO on cellulose acetate stick at room temperature. The band at 2130 cm\(^{-1}\) assigned to CO stretching (\( \nu_{\text{CO}} \)) indicated CO adsorption on cellulose acetate stick after exposure to CO (Wang et al. 2017). The intensity of the band became stronger with the adsorption time until it reached CO adsorption equilibrium at around 8 min. The normalized CO adsorption amount remained unchanged after 8 min.

The in-situ DRIFTS spectra of CO adsorbed on the cellulose acetate stick surface for reaching CO adsorption equilibrium in the temperature range from 25 to 300 °C were further organized, as shown in Fig. 7. It can be seen that the CO saturated adsorption capacity decreased with the increase of adsorption temperature. When the adsorption temperature reached 300 °C, the amount of adsorbed CO could be neglected. Combined with the infrared thermal imaging results, we concluded that the CO adsorption occurred only in the partial region of cellulose acetate stick where the temperature was between 25 and 300 °C, while, there was no CO adsorption in the region of cellulose acetate stick where the temperature was beyond 300 °C.

The integrated areas of the characteristic bands of \( \nu_{\text{C}-\text{O}} \) as a function of irradiation time under different temperatures are shown in Fig. 8. At each temperature point, the change of intensities of asymmetric and symmetric of \( \nu_{\text{C}-\text{O}} \) reached a steady level after 8 min approximately. As the temperature is increasing from 25 to 300 °C, the CO saturated adsorption gradually decreases, because the high temperature is not conducive to the adsorption of gas (Yang et al. 2021).

Besides CO, formaldehyde, acetone, and propylene glycol as the typical smoke substances are also harmful to human health, and their adsorption ability on the surface of the cellulose acetate stick are also greatly affected by the adsorption temperature (Alalwan and Alminshid 2020). The adsorption characteristic of such smoke stream substances on the surface of cellulose acetate stick were observed by in-situ DRIFTS using the same approach as CO adsorption. The breakthrough curves of the formaldehyde adsorption, acetone adsorption and propylene glycol adsorption versus adsorption time at different adsorption temperatures on cellulose acetate stick are shown in Fig. 9a–c, respectively. The result revealed that the formaldehyde, acetone, and propylene glycol adsorption characteristics followed the same trend as CO adsorption. Therefore, it can be concluded that the sorption capacities of cellulose acetate stick for all the typical smoke stream substances are all dependent on
the adsorption time and temperature. At the same adsorption temperature, the adsorption abilities of smoke substances increased with the adsorption time until the surface adsorption of cellulose acetate stick was saturated. The relatively higher speed of adsorption in the beginning of the adsorption process can be connected with the high amount of free active sites which can be filled with smoke substances. Meanwhile, the adsorption characteristics of all these typical smoke stream substances fit well with the Freundlich model (Oh et al. 2020). It is worth noting that the adsorption ability of the cellulose acetate stick for different type of typical smoke substances is different because of the differences in chemical properties and molecular size of smoke substances (Xiao et al. 2021). In particular, the propylene glycol adsorption affinity of cellulose acetate stick is greatest among the smoke substances with the lowest adsorption equilibrium point due to the closest polarity to cellulose acetate (Davydov and Posrednik 2020). Based on the obtained data, it could be concluded that the absorption abilities of smoke substances decrease with the adsorption temperature at the same adsorption time, as the high temperature is not conductive to the
absorption of smoke substances on the surface of cellulose acetate stick.

In order to observe the adsorption ability of cellulose acetate stick for typical smoke stream substances changes with the temperature/space coordinate intuitively, the transformation of the data of the breakthrough curves is required. Take the CO adsorption as an example, the experimental results of CO adsorption presented in this work allowed for a mathematical model to be established [Eq. 3], describing the normalized CO adsorption amount versus the different temperatures. The corresponding mathematical model was obtained by fitting the data in Fig. 10 with the equation as follows:

$$\text{CO}_{\text{adsorption}} = -2.63E^{-9}T^3 + 5.62E^{-6}T^2$$

$$- 0.00363T + 0.668$$

(3)

The reason for acquiring and mathematically modeling the data presented in Fig. 10 is that it can be used to predict and calibrate the CO adsorption ability at any temperature in the range of 25–930 °C. Therefore, this mathematical model is significant to predict CO dynamic variations in the whole cellulose acetate stick. Specifically, combined the above mathematical model [Eq. 3] with the temperature gradient model equation along the $r$-axis [such as Eq. 1], we can easily get the CO adsorption characteristics along the radial direction in the 2D cross-sections; take the 2D cross-section at $x = 10$ mm for example. The fitting curves of normalized CO adsorption for the 2D section at $x = 10$ mm acquired by the mathematical treatment with Figs. 5b and 10 are shown in the Fig. 11a, the corresponding equations of the curves that fitted are as followed:

$$y = -2.63E^{-9}x^3 + 5.62E^{-6}x^2 - 0.00363x + 0.668$$

$$R^2 = 0.997$$

Fig. 9 Breakthrough curves of a formaldehyde, b acetone and c propylene glycol adsorption versus adsorption temperature on the cellulose acetate stick

Fig. 10 The fitting curve for CO adsorption ability versus adsorption temperature after CO adsorption saturation, insertion is the fitted polynomial equation
After the CO adsorption characteristics of all the 2D cross-sections were identified, we could calculate the average CO adsorption ability at different 2D cross-sections by the integral method. Then, the information on the average CO adsorption along the different x-axis was acquired (Fig. 11b) and fitted in the same way as the Eq. 2:

\[
\text{CO}_{\text{adsorption}} = 0 \ (0 \leq x \leq 4) \quad (6)
\]

\[
\text{CO}_{\text{adsorption}} = -0.001x^3 + 0.032x^2 - 0.234x + 0.5045 \ (4 \leq x \leq 16) \quad (7)
\]

\[
\text{CO}_{\text{adsorption}} = 0.589 \ (16 \leq x \leq 20) \quad (8)
\]

As shown in Fig. 11b, the total CO adsorption capacity of the cellulose acetate stick could be roughly calculated by the integration, yet, it was not possible to intuitively analyze the dynamic alteration of CO adsorption. In order to solve this problem, Fig. 12 shows the 3D model of the normalized CO adsorption of cellulose acetate stick versus the spatial coordinates. The normalized CO adsorption can be observed at any point in the cellulose acetate stick. Almost no CO adsorption is observed in the area near the tobacco burning side due to their higher temperature (> 300 °C). Then the CO adsorption ability gradually increased both along the x-axis and r-axis until it was saturated as the temperature gradually reduced. Compared with Fig. 11b, we could exactly calculate the total amount of adsorbed CO in whole cellulose acetate stick with the 3D adsorption model (Fig. 12) by the integral method at any time point. Based on this, the dynamic distributions of the typical smoke stream substances could also be modeled with the normalized adsorption amount of versus the time. With it, the amount of typical smoke stream substances released by cigarettes after the interception of cellulose acetate stick could be controlled. The CO-TPD experiments also detailed the adsorption and retention characteristics (Fig. S2). Before desorption measurement, CO adsorption was conducted at 50 °C. An evident peak appeared in the pattern, which could be assigned to the characteristic peak of physical adsorption of CO. To some extent, the effective desorption indicated that the stick was effective in retaining CO at low temperatures.

Conclusions

Although aromatic hydrocarbons and smoke tar may be more toxic than low molecular carbon such as CO, formaldehyde, propylene glycol and acetone, the adsorption of the low molecular carbon substances on the cigarette filter are more sensitive to the temperature than those of the aromatic hydrocarbons and smoke tar. Therefore, in such situation, we chose the low molecular carbon substances as the adsorption objects. A combined in-situ DRIFTS and the infrared thermal imaging method were established and applied to investigate the adsorption of CO and typical low-carbon aldehydes, formaldehyde, propylene glycol, and acetone, on cellulose acetate stick. The 3D temperature distribution with the fitting curves of temperature versus x-axis and r-axis was obtained by
the mathematic fitting with the infrared thermal images results for confirming the adsorption temperature range, which was used to perform in-situ DRIFTS experiment in accurate temperature conditions. The adsorption capacities of cellulose acetate stick to these typical smoke stream substances were correlated with the adsorption time and temperature, and the corresponding models equations in cellulose acetate stick were established. The adsorption characteristic of the cellulose acetate stick for different smoke substances was different because of the differences in chemical properties and molecular size of smoke substances. The propylene glycol adsorption was strongest among smoke stream substances. By combining the 3D temperature distribution model with the adsorption model equations, the 3D model of adsorption capacity versus the spatial coordinates was established for intuitively illustrating the dynamic distributions of the typical smoke stream. Future research on the adsorption characteristics of the cigarette filter for aromatic hydrocarbons and smoke tar may extend the understanding of smoke adsorption and retention rules on the cigarette filter for the purpose of effectively controlling smoke emissions and reducing the toxic effect of smoking on human health. The information obtained in this work is useful for guiding analytical pyrolysis studies aimed at assessing precursor-smoke toxicant relationships and the fate of tobacco ingredients added to cigarettes.

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Declarations

Conflict of interest Authors declare no conflicts or competing interests.

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