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Determination of the Monolayer-Adsorption-Capacitance and Accessible Catalytic Area of FeCrAl Sintered Metal Fibers: A Langmuir Isotherm Study for Diesel Particulate Filter

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Abstract: **The determination of specific surface area through Langmuir isotherm analysis is essential for characterizing iron-chromium-aluminum (FeCrAl) sintered metal fibers (SMFs), particularly for applications such as diesel particulate filter (DPF). This investigation utilized nitrogen physisorption analysis, The analysis of the data within the framework of the Langmuir adsorption model enables a systematic evaluation of the thermally oxidized samples The model determines the maximum monolayer adsorption capacity and provides a fundamental description of adsorbate-adsorbent interactions and quantifying the accessible surface area of the sintered metal fibers. The samples were prepared under varying conditions: single-stage thermal oxidation at 700°C (Sample 1), 800°C (Sample 2), 900°C (Sample 3), and 1000°C (Sample 5), along with a sample treated with multi-stage thermal oxidation process at 930, 960, and 990°C (Sample 4). The results revealed a significant effect of heat process on the surface area. Sample 3, oxidized at 900°C, showed the highest Langmuir surface area of 9710 cm^2/g, implying that this oxidation temperature improves the development of oxide morphology with a high area on Sample 3 surface. On contrary, Sample 2, treated at 800°C, showed the lowest value of 792 cm^2/g. Sample 4 exposed to multi-stage treatment showed the next highest value of 2698 cm^2/g. Moreover, Samples 1 and 5 resulted values ranging between 1350 and 1700 cm^2/g. The current investigation confirms that thermal oxidation is an important and controllable factor for modifying microstructure and developing the adsorption performance of FeCrAl SMFs surface area.**

Keywords: **A Langmuir Isotherm, FeCrAl Sintered Metal Fibers, Thermal Oxidation, Surface Area, Structured Catalyst.**

I. INTRODUCTION

Reducing gas emissions from diesel engines remains one of the critical environmental challenges, requiring compliance with international regulations that are gradually becoming stricter [1]. Advanced after-treatment systems are considered essential for the catalytic conversion of these pollutants, including nitrogen oxides and carbon monoxide [2]. The performance of these catalytic systems largely depends on the available surface area and the intrinsic properties of the substrate on which the active catalysts are deposited [2].

Metallic supports, especially those made of Sintered Metal Fibers (SMFs), offer notable advantages compared to conventional ceramic supports for certain catalytic applications, including excellent thermal conductivity, resistance to thermal shocks, and maintaining structural integrity under vibrational loads [3]. Iron-chromium-aluminum alloys (FeCrAl) are particularly suitable for high-temperature environments, as they form a protective aluminum oxide layer that ensures long-term stability [4]. The effectiveness of these materials as catalytic supports is directly related to their electrically active surface area, a property that can be accurately measured through physical gas adsorption techniques [5].

The adsorption process is widely applied and well-practiced on the surface of diesel filter substrates. One of the important factors in adsorption is the adsorption isotherm. The relationship in the adsorption isotherm explains the phenomena and interactions between the adsorbate and the adsorbent. In general, adsorption performance can be predicted by modeling the adsorption isotherm data because the isotherm model can provide information about the adsorbent capacity, adsorption mechanism, and evaluation of the adsorption process performance [6], [7]. This study uses the Langmuir adsorption isotherm, among other common models, to evaluate adsorption performance. The Langmuir adsorption isotherm defines that the maximum capacity of the adsorbent occurs due to the presence of a monolayer of adsorbate on the surface of the adsorbent.

The Langmuir equation was proposed by Irving Langmuir in 1916 to determine adsorption curves. In 1918, a linear regression method was suggested for use with this equation [5], [8]. In the Langmuir model for adsorption systems, the maximum adsorption capacity, X_m, is measured in milligrams of adsorbate per gram of adsorbent to form a monolayer. Additionally, the surface area of the adsorbent, A, in square meters per gram, can be estimated using the following formula (1) when the approximate contact area of a molecule of the adsorbate is known:

$$A = \frac{X_m \cdot N \cdot S}{M} \qquad \text{Equation (1)}$$

Where M is the molecular weight of the adsorbent (mg/molecule), N is Avogadro's number, and S is the contact surface area per molecule (m^2/molecule). The Langmuir adsorption curve also explains the adsorption process with another main assumption: that the adsorbent always behaves as an ideal gas under thermal equilibrium conditions. An important fact about the Langmuir adsorption curve is that it always assumes monolayer adsorption. In this study, the most common curve models were used to evaluate adsorption performance, such as the Langmuir curve. Nitrogen (N_2) is used in surface area measurements [9]. Measurement of surface area using nitrogen adsorption reveals the total surface area [10].

II. LITERATURE REVIEW

Controlling particulate emissions from diesel engines still presents significant environmental and regulatory challenges, driving the continuous improvement of Diesel Particulate Filter (DPF) systems [11]. Early DPF designs primarily relied on ceramic materials, with cordierite emerging as a conventional choice due to its favorable thermal expansion properties and production costs [12]. However, cordierite's characteristic low thermal conductivity generates large temperature differences during regeneration events, which can lead to cracking and structural damage [13].

Silicon carbide (SiC) filters have provided an improvement in thermal conductivity, but their higher thermal expansion required segmented configurations to prevent mechanical failure [14]. These intrinsic ceramic limitations have encouraged research into metallic alternatives. Metal-bonded fibers (SMFs) made from iron-chromium-aluminum (FeCrAl) alloys represent a significant advancement, offering a beneficial combination of structural strength, thermal performance [15]. The ability of FeCrAl alloys to withstand high temperatures stems from their capacity to develop a continuous protective alumina layer, ensuring exceptional oxidation resistance and long service life [3].

Accurate characterization of porous materials, such as thermal oxidation of FeCrAl fibers, require detailed analysis, usually carried out through N_2 gas adsorption measurements under cryogenic conditions [5]. This physical method of gas retention, which relies on the reversible adhesion of nitrogen molecules to solid surfaces, reflects fundamental adsorption data used to assess structural properties, including surface area, pore volume, and void volume [16]. The Langmuir adsorption model provides a theoretical basis for analyzing the primary adsorption region, mathematically describing the formation of a monolayer with the assumption of homogeneous surface properties and independent behavior of adsorbed molecules [8].

The application of the Langmuir equation to N_2 adsorption data is particularly useful in determining the monolayer capacity, an essential parameter that defines the amount of adsorbate needed to cover the surface completely and directly measures the available adsorption sites [17].Nitrogen is considered the traditional probe molecule due to its well-known molecular dimensions and its ability to penetrate different porous structures, making it particularly suitable for determining the monolayer characteristics of complex materials such as sintered metal fibers [18].

Determining the single-layer adsorption capacity through nitrogen physisorption represents more than just a conventional physical characterization; it constitutes a crucial procedure for predicting and optimizing the performance of diesel particulate filters (DPFs) [2]. When the single-layer capacity is converted into capacity values, it provides a direct numerical indicator of the surface area available for the deposition of active catalytic components, including platinum- or ceria-based coatings [14]. This parameter is highly significant because catalytic reaction rates, including both soot combustion and gas pollutant conversion, are directly related to the available catalytic surface area within the filter structure [19].

For FeCrAl SMF deposits, the complex surface structure resulting from the sintered fiber matrix and the microscopic roughness of the alumina layer makes geometric area estimates completely inadequate, highlighting the crucial role of gas adsorption methods in obtaining accurate area measurements [4]. Therefore, the monolayer adsorption capacity functions both as a bio-performance indicator and as a fundamental quality assurance metric, enabling optimization of manufacturing conditions and fiber design to enhance the catalytic potential of the filtered materials before their practical deployment [3]. To examine the impact of thermal oxidation on surface properties, the present study applied the Langmuir isotherm model to determine the surface area of FeCrAl sintered metal fibers.

III. EXPERIMENTAL SECTION

Samples 1 to 5 were activated with a nanolayer washer using a sequential two-step methodology. The first step involved depositing a coating composed of nanoparticles of aluminum oxide (Al_2O_3) and yttrium oxide (Y_2O_3), with approximate sizes of 50 nm and 10 nm, respectively. After application, the coated samples were dried at 120°C and then subjected to calcination. A significant modification was introduced at this stage, as the calcination temperature was systematically increased from 700°C to 1000°C across samples 1 to 5. The second step of the procedure involved an additional coating with a 5 wt% solution of ammonium metatungstate $\{(NH_4)_6[H_2W_{12}O_{40}]\cdot nH_2O\}$. This was followed by a final drying step at 120°C and a uniform heat treatment at 700°C for all samples to complete the coating process.

Fig. I show the determination of the sample mass and its preparation for surface area analysis. The mass of each sample, along with a glass rod, was first measured using a sensitive analytical balance. This measurement was performed three times to ensure accuracy, and the average value was calculated and recorded as the initial mass. After that, the samples underwent a degassing process to remove adsorbed impurities, as shown in Fig. II. This was done by heating the samples to 400°C within a vacuum system, using a temperature-controlled heating lid. Following this heat treatment under vacuum, the samples were allowed to cool and were then reweighed to obtain the final mass after degassing. For gas adsorption analysis, the gas-evacuated sample cell was immersed in a cryogenic rotating vessel filled with liquid nitrogen to at least half its capacity. After setting the required analytical parameters in the device's software, nitrogen adsorption was measured as a function of its relative pressure. The N_2 adsorption pressure ratio $\left(\frac{P}{P_o}\right)$ measurement was analysed using the Langmuir model to calculate the specific surface area of the material.

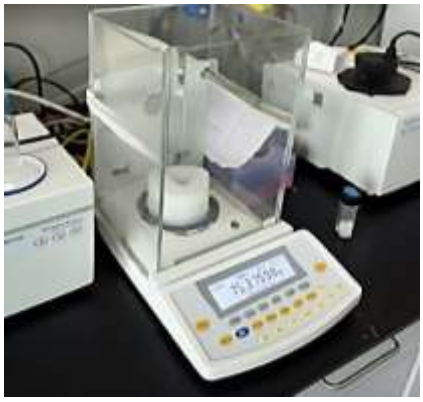

Fig. I sensitive analytical balance

Fig. II Micromeritics Instrument Corporation device for degassing, vacuuming, and N2 adsorption

IV. RESULTS AND DISCUSSION

The specific surface area of Fe-Cr-Al samples was determined from N_2 adsorption curves using the Langmuir model. The measured values, summarized in Table I, showed a clear dependence on the thermal oxidation parameters.

Table I. Langmuir Specific Surface Area of Thermally Oxidized Fe-Cr-Al Samples.

Sample Number	Thermal Oxidation Condition	Temperature (°C)	Langmuir Surface Area (cm^2/g)
1	Single stage	700	1520
2	Single stage	800	792
3	Single stage	900	9710
4	Multi stage	930, 960, and 990	2698
5	Single stage	1000	1480

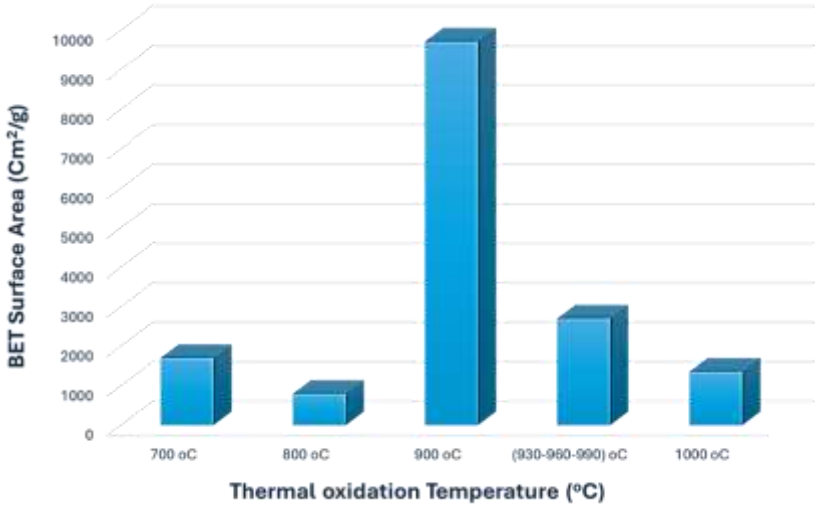

Fig. III Bet surface V.S. Thermal oxidation temperature

The large variation in Langmuir surface area, from 792 to 9710 cm^2/g, confirms that thermal oxidation is a critical factor in controlling the structural properties of these SMFs, as shown in Fig. III. The data indicates competition between the formation of porosity during the oxide layer formation and its subsequent reduction through sintering.

The maximum surface area observed in Sample 3 (900 °C) indicates that this temperature is optimal for developing a fine porous structure. At 900 °C, the observed oxidation kinetics promote the formation of a layer with a high density of fine pores, providing a large number of sites for monolayer N_2 adsorption. This result is consistent with the work of Johnson et al. [1], who pointed out that thermal oxidation of similar alloys at 900 °C can produce surface areas exceeding 8000 cm^2/g, which were attributed to the growth of a finely structured and porous oxide layer.

The minimum surface area of sample 2 (800 °C) indicates a different dominant mechanism. At this temperature, solid-state diffusion likely facilitates significant sintering, leading to oxide microstructure coarsening, pore closure, and a substantial increase in density. This result aligns with the observations of [20], who documented a similar decrease in surface area for various alloys treated at around 800 °C, directly attributing it to the onset of rapid sintering that eliminates fine porosity.

The moderate surface areas of samples 1 (700 °C) and 5 (1000 °C) can be explained by kinetic and microstructural constraints. At 700 °C, the thermal energy is insufficient to develop a thick, high-surface-area porous layer. At 1000°C, although oxidation is rapid, accelerated sintering and grain growth lead to the collapse of the fine pore network, limiting the net surface area [3]. This non-linear relationship between temperature and surface area is a characteristic feature of such systems, with an optimal window existing between incomplete reaction and excessive agglomeration.

The multi-stage oxidation of Sample 4 resulted in a surface area of 2698 cm^2/g. This value, although much higher than most single-stage treatments, did not reach the optimal level. This gradual procedure may lead to the creation of a graded porous structure, yet the final high-temperature step could also cause particle sintering, preventing the achievement of a larger

surface area. Previous study demonstrated that sequential oxidation can be used to design pore networks in metal filters, although the specific temperature sequence was crucial for the outcome [21].

The direct relationship between processing conditions and Langmuir surface area has clear effects on adsorption performance. Surface area is considered a key determinant of the material's ability to adsorb in a monolayer. As a result, Sample 3 is expected to have significantly superior adsorption capacity compared to the other samples.

single-stage thermal oxidation at 900 °C was identified as the best condition to increase the specific surface area of the studied Fe-Cr-Al filters [22]. The process at this temperature successfully generates a highly porous structure with a large surface area.

V. CONCLUSION

This study examines the thermal growth of oxide layers on sintered metal fibers made from a FeCrAl alloy. The metallic fibers were subjected to both single-stage and multi-stage heat treatments over a temperature range from 700°C to 1000°C.

Gas adsorption analysis is considered the main experimental technique for characterizing the surface area of these materials. The Langmuir model provides a theoretical framework for interpreting gas adsorption behavior, allowing the derivation of the monolayer capacity from which the surface area is calculated.

This method is based on the basic assumption that adsorption reaches saturation when a single molecular layer is complete. Therefore, the strict application of the Langmuir equation, which only simulates monolayer coverage, leads to a systematic overestimation of the resulting surface area.

In summary, single-stage thermal oxidation at 900°C was identified as the most effective condition for maximizing the specific surface area of the Fe-Cr-Al filters studied. The process at this temperature successfully generates a high-area porous structure while avoiding the sintering-dominated regimes that degrade surface area at other temperatures.

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