



INTRAMICRON

Sulfur Sorbent

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IntraMicron has developed various types of sorbents for removing sulfur species, volatile organic compounds (VOCs), ammonia, etc. from liquid or gas streams. Most of these sorbents are supported on supports such as activated carbon, silica, alumina, and titania, which have high surface areas ($>100 \text{ m}^2/\text{g}$) and pore volumes ($\sim 1 \text{ cm}^3/\text{g}$). Thus, most sorbents realize enhanced utilization of active compounds and can be regenerated for multiple usage cycles. Here, desulfurization sorbents will be used as an example to demonstrate the sorbent development capabilities at IntraMicron.

The sorbent for gas phase sulfur removal is made of transition metal (such as copper, silver, iron, etc.) promoted ZnO supported on silica particles (60-80 mesh). Due to the support and active phase structure as well as the dopant, intra-particle mass transfer and lattice diffusion become extremely fast even at low temperatures (i.e. room temperature). As a result, the sorbent has high ZnO utilization and high sulfur capacity (Table 1). For the same reason, the sorbent can be regenerated at lower temperatures and used for multiple cycles, as shown in Figures 1 and 2.

Table 1. Sulfur capacities of several doped ZnO/SiO₂ sulfur sorbents. The dopant loading is 5 wt % of the ZnO

Dopant	Saturation Capacity		Breakthrough Capacity (g S / g ZnO)
	(g S / g ZnO)	% Utilization	
CuO	0.213	54	0.163
Ag ₂ O	0.189	48	0.163
Ce	0.177	45	0.142
CuO-La ₂ O ₃	0.161	41	0.122
La ₂ O ₃	0.140	35	0.122
MnO _x	0.132	33	0.102
NiO	0.113	29	0.102
CoO _x	0.113	29	0.081
Undoped	0.113	29	0.081

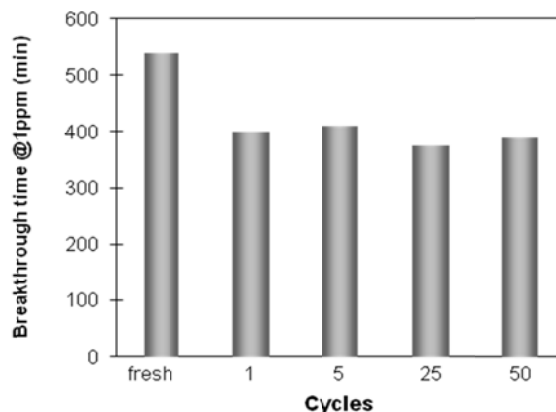


Figure 1. Breakthrough times of ZnO/SiO₂ sulfur sorbent regenerated at 550°C.

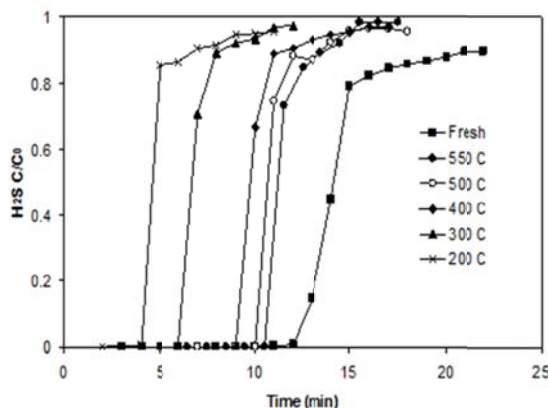


Figure 2. Breakthrough curves following regeneration of the sulfur sorbent as a function of temperature.

During regeneration, no special media such as inert gas or reducing agents are required. Air or an air-steam mixture can easily recover sulfur removal capacity. Most commercial sulfur sorbents require a regeneration temperature of 600 °C, with regeneration efficiencies less than that of IntraMicron's sorbents (Figure 3). The sorbent developed by IntraMicron can be regenerated at 550 °C because of its highly porous structure. After the first regeneration cycle, its sulfur capacity drops to 80% of the

original capacity, but this capacity is stable for at least 50 high-temperature regeneration cycles (Figure 1). Low temperature regeneration is also feasible. Although sorbent capacity recovery is less effective at lower temperatures, most of the sorbents capacity can still be recovered at temperatures exceeding 300 °C. The balanced desulfurization performance and the ease of operation make this sorbent a superior candidate for applications in mobile fuel cell systems in terms of reduced reactor weight and volume, low cost, process convenience, and ease of integration. This sorbent is especially suited to desulfurization applications for PEM fuel cells and solid oxide fuel cells, even during cold startup.

In addition to gas-phase desulfurization sorbents, IntraMicron has also developed sorbents designed to remove sulfur from liquid fuels such as LPG, gasoline, and jet fuels at room temperature. As a milestone for liquid fuel desulfurization, these sorbents provide the following advantages: (1) high sulfur removal capacity (6~15 mg sulfur/g of adsorbent) for jet fuels, perhaps highest among regenerative adsorbents previously developed (Figure 4); (2) ease of regeneration, which requires only air and low temperature heat and no special regeneration media (e.g. sulfur free hydrocarbons, high purity nitrogen, helium, etc.); (3) much larger particle size than other previous formulations, resulting in almost negligible pressure drop while on stream and (4) low cost due to the use of well dispersed, low concentration silver on TiO₂, which precludes the use of precious metals or specially prepared supports.

The sorbent can be entrapped in microfibrus media for improved performance (see microfibrus entrapped catalysts and sorbents). For regenerable desulfurization applications, micron-sized glass fibers are used to entrap IntraMicron's sorbents to maintain effectiveness during both the highly corrosive desulfurization operation as well as the excessive oxidation encountered during regeneration. Glass fibrous entrapped sorbents, as depicted in Figure 5, can further improve the heterogeneous contacting efficiency between the gas stream and sorbent particles. One important application of glass fibrous entrapped sorbents is utilization as a thin polishing layer at the downstream end of a typical packed bed, and this combined approach is referred to as the composite bed. Without significantly increasing bed length, the breakthrough time of the composite bed can be 3 times longer than that of the packed bed (Figure 6).

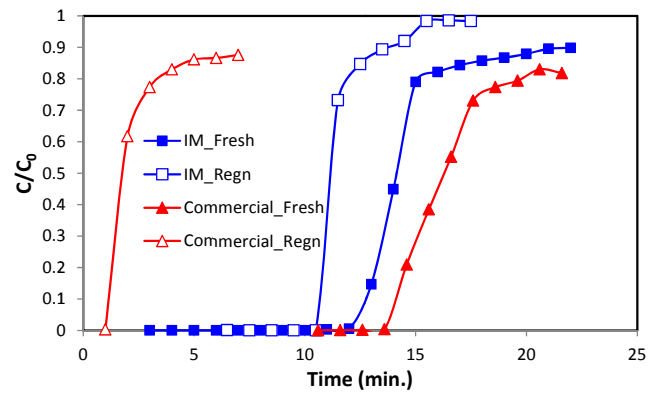


Figure 3. Breakthrough comparison of IntraMicron's sorbent with a commercial H₂S Sorbent before and after regeneration.

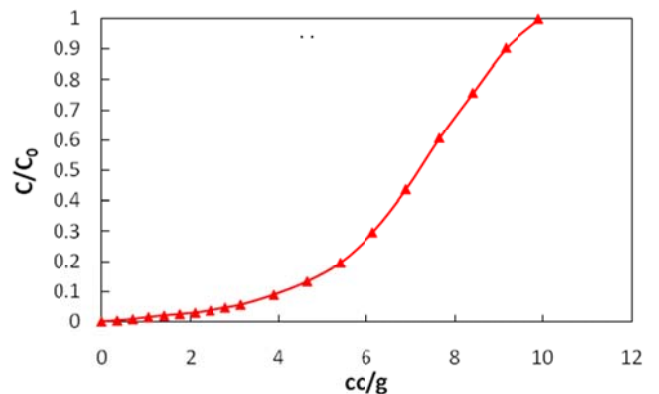


Figure 4. Breakthrough curve for jet fuel desulfurization. Seven grams of sorbent tested with JP 5 containing 1172 ppmw of sulfur at a flow rate of 0.5 cc/min.

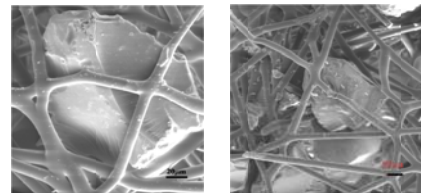


Figure 5. Glass-fiber-entrapped sulfur sorbent.

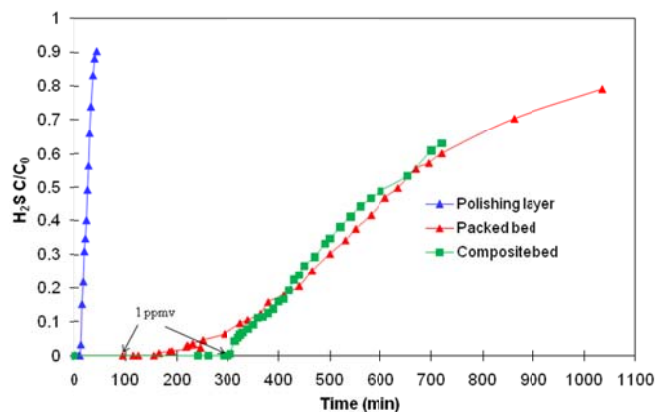


Figure 6. Using a composite bed with a microfibrus-entrapped polishing layer triples the H₂S breakthrough time.