Optimisation of Post-Treatment Conditions for Improved Coating Adhesion in Structured Catalysts

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INTRODUCTION

Achieving a stable and durable catalyst coating is essential for structured catalysts in CO_2 hydrogenation to methanol. While surface pre-treatment and slurry optimisation improve coating quality, **post-treatment conditions,** particularly calcination, directly impact adhesion stability [1]. Post-treatment, especially calcination, plays a vital role in stabilizing the coated catalyst layer by influencing **thermal expansion, phase transformation, and coating morphology** [2]. The heating process removes residual solvents, promotes phase stabilisation, and enhances **mechanical integrity**, ensuring a **well-adhered** catalytic layer [3].

OBJECTIVE

This study aims to evaluate the impact of **calcination ramp rates on coating adhesion stability.** The investigation focuses on identifying the relationship between heating rate and coating durability, assessed through **ultrasonic adhesion testing** to quantify coating retention and weight loss.

METHODOLOGY

1. Sample Preparation

– Previously **optimised Cu/ZnO/Al₂O₃ slurries** were used for coating on the **surface improved FeCrAlloy® foil** substrate.

– Slurries from previous work are:

Slurry ID	Slurry composition
SL02	15 wt.% Cu/ZnO/Al₂O₃ , 2 wt.% polyvinyl alcohol, + 7 wt.% Col. Alumina
SL06	15 wt.% Cu/ZnO/Al₂O₃ , 2 wt.% polyvinyl alcohol, + 7 wt.% Col. Silica
SL07	15 wt.% Cu/ZnO/Al₂O₃ , 2 wt.% polyvinyl alcohol, + 7 wt.% Col. Zinc
SL08	15 wt.% Cu/ZnO/Al₂O₃ , 2 wt.% polyvinyl butyral, + 7 wt.% Col. Alumina
SL09	15 wt.% Cu/ZnO/Al₂O₃ , 2 wt.% polyvinyl butyral, + 7 wt.% Col. Silica
SL10	15 wt.% Cu/ZnO/Al₂O₃ , 2 wt.% polyvinyl butyral, + 7 wt.% Col. Zinc

*Col. - colloidal

– Two samples were prepared using each slurry.

– **Samples** underwent calcination at two different heating rates **(2°C/min and 10°C/min)** to analyse post-treatment effects.

2. Coating Characterisation

– % Loading (I)

Sample weights were measured during each phase of the process and Calculated as:

$$\% \; loading \; (l) = \frac{m_{wash} - m_{sub}}{m_{sub}} \times 100\%$$

Where m_{wash} is the weight of the coated sample, and m_{sub} is the weight of the uncoated sample.

– Ultrasonic Adhesion Test

Conducted in an ultrasonic bath (SONOREX super 535, bandelin) operating at 35 hz for 30 minutes, with samples immersed in petroleum ether to evaluate coating adhesion stability as **weight loss (%)** using:

$$w_{ultra} = \frac{m_{wash} - m_{ultra}}{m_{wash} - m_{sub}} \times 100\%$$

Where w_{ultra} is the weight loss on ultrasonic vibration, and m_{ultra} is the weight of the sample after the ultrasonic test.
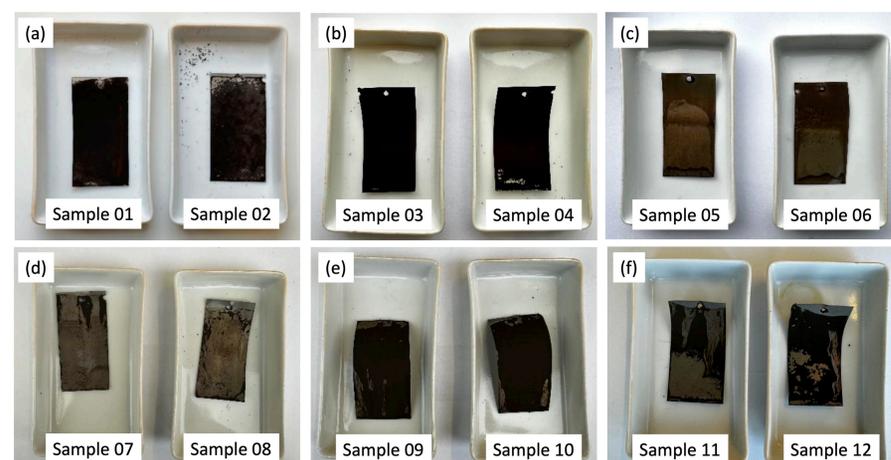
ANALYSIS RESULTS

Figure 4.13: Digital image of dip-coated substrate with slurry (a) SL02, (b) SL06, (c) SL07, (d) SL08, (e) SL09, and (f) SL10

Effect of Calcination Ramp Rate on % loading and % weight loss

Sample ID	Slurry used	Heating rate	% Loading (I)	% Weight loss (w_{ultra})
01	SL02	2	5.32	3.24
02	SL02	10	8.78	59.81
03	SL06	2	4.33	2.23
04	SL06	10	4.17	23.48
05	SL07	2	2.15	5.47
06	SL07	10	2.03	31.67
07	SL08	2	1.52	58.33
08	SL08	10	1.04	89.09
09	SL09	2	15.61	64.29
10	SL09	10	8.93	95.625
11	SL10	2	6.30	89.14
12	SL10	10	7.89	93.50

Coating detachment was observed in some 10°C/min samples even before the adhesion test, with catalyst residue found on the tray inside the muffle oven. No visible microcracks or cracks were detected by the naked eye after calcination.

KEY FINDINGS

Coating Quality Assessment

– The best coating results were achieved with **SL02 (PVA + col. alumina)** and **SL06 (PVA + col. silica),** which formed homogeneous coating layers.

– SL09 (PVB + col. silica) formed a thick coating, but the coating was not uniform.

– SL07 (PVA + col. zinc) resulted in a very thin coating layer.

Adhesion Test Findings (ultrasonic vibration at 35 kHz for 30 min)

– **SL02 (PVA + col. alumina)** and **SL06 (PVA + col. silica)** at 2°C/min demonstrated the best adhesion, with weight losses of **3.24%** and **2.23 wt.%,** respectively both within the target weight loss threshold of **<5 wt. %.**

– **SL07 (PVA + col. zinc)** resulted in a **weight loss of 5.47%,** slightly above the threshold.

– SL09 (PVB + col. Silica) exhibited high catalyst loading but poor adhesion, leading to high weight loss after ultrasonic testing.

RESEARCH OUTLOOK

Further characterisation with XRD, SEM and chemisorption technology. Scaling up to monolithic structured catalyst. Application and testing in reactor facility to investigate the improvement in catalyst performance. conditions.

REFERENCES
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