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Focus on the Thermoadhesion Mechanism in the Formation of Fayalite-Type Ceramic Deposits in Particle Separators of Fluidized-Bed Reactors

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Abstract. This study examines the thermoadhesive mechanism responsible for the formation of fayalite-type ceramic deposits in particle separators within fluidized-bed reactors. The analysis focuses on the influence of temperature, pressure, and collision-induced energy on the changes in viscosity and plasticity of the material system. Particular attention is given to operational conditions within the temperature range of 450 °C to 750 °C and pressures exceeding 5 bar. The findings suggest that deposit formation occurs when the energy released during particle collisions increases the local temperature of the particles, thereby reducing their relative viscosity and enhancing their plastic behavior. This transition promotes adhesion and the subsequent accumulation of material on the internal surfaces of the equipment. Furthermore, mathematical modeling, incorporating a modified version of McClean's creep theory, is employed to estimate the critical conditions for deposit formation. These models provide valuable insights for optimizing process design and operational control in industrial systems involving ceramic materials.

Keywords: adhesion, particle collision, thermos-adhesion, relative viscosity, wustite.

Enfoque en el mecanismo de termoadhesión en la formación de depósitos cerámicos de tipo fayalita en separadores de partículas de reactores de lecho fluidizado

Resumen. Este artículo analiza el mecanismo termo-adherente que conduce a la formación de acumulaciones de fayalita en separadores de partículas dentro de reactores de lecho fluidizado. La investigación se centra en cómo factores como la temperatura, la presión y la energía liberada en las colisiones entre partículas influyen en el cambio de viscosidad y la plasticidad del sistema, particularmente en rangos de temperatura de 450 °C a 750 °C y presiones superiores a 5 bar. Se propone que estas adherencias se forman cuando la energía de impacto calienta las partículas, disminuyendo su viscosidad relativa y favoreciendo su viscosidad plástica, lo que facilita su adherencia y acumulación en las superficies internas del equipo. La utilización de modelos matemáticos, basados en la modificación de la teoría de McClean para la termofluencia, permite estimar las condiciones que generan estas adherencias, así como su potencial aplicación en el diseño y control de procesos cerámicos similares.

Palabras clave: adherencia, choque de partículas, termo adherencia, viscosidad relativa, wustita

I. INTRODUCTION

During the different iron ore manufacturing processes in which fluidized bed technology is employed for material processing [1], these systems tend to operate under an intermittent regime. This intermittent behavior is primarily caused by the phenomenon of de-fluidization, which occurs due to the adhesion of partially reduced iron particles within the fluidized bed [2]. This de-fluidization is closely linked to the formation of accretions on the internal metallic surfaces of cyclones. Such accretions result from repeated collisions between particles at temperatures below the melting point of iron, thereby interrupting the continuous flow and stability of the process.

In this context, the concept of particle interaction is associated with the phenomenon of apparent viscosity [3], which quantitatively expresses the resistance of iron-containing particles to creep movement under plastic deformation. This concept encompasses multiple factors, including external frictional interactions, cohesive forces such as Van der Waals forces, interfacial attractions, and liquid–solid bridges, among others. However, these factors have not been fully integrated into current models describing particle agglomeration phenomena in fluidized bed processes.

To better explain the formation of silicate adhesions within mechanical particle separators, this study proposes a *thermoadhesion approach*. This approach posits that the energy released from the collision and impact of particles on the separator's internal surfaces generates localized heating. This, in turn, induces changes in the viscosity of particulate materials, leading to a transition to a plastic state that favors adhesion and accumulation on these surfaces.

Rheology, the branch of physics concerned with the deformation and flow behavior of matter — ranging from simple liquids and solids to complex microstructured systems such as silicates — provides the theoretical foundation for analyzing this process. Of particular interest are materials whose viscosity varies as a function of the deformation rate within a specific temperature range. These are known as non-Newtonian fluids.

To address this apparent complexity, rheology is employed as it allows the description of flow behaviors where viscosity is dependent on the rate of deformation. This theoretical framework links the flow behavior of a material to its internal structure. For materials like silicates, this relationship cannot be fully described using classical fluid mechanics or elasticity alone, thus requiring more advanced rheological models to capture their behavior accurately.

II. DEVELOPMENT

The phenomenon under study occurs within a particle separator of a fluidized bed reactor during the reaction between ferrous oxide (FeO) and metallic iron (Fe). This reaction takes place within a temperature range of 450 °C to 750 °C and at pressures exceeding 5 bar. Under these operating conditions, the mechanism involves the adhesion of materials, primarily FeO and SiO_2, induced by the energy released from particle collisions.

The proposed mechanism considers the behavior of rotating particles within the particle separator. It is supported by mathematical formulations designed to describe the phenomena in terms of apparent viscosity and the resulting plasticity. These parameters were evaluated through the analysis of adhesion samples with a high concentration of ferrous oxide, which were collected from industrial processes operating under similar conditions.

To characterize these adhesions, Bauman print-type macroetching techniques were employed. This method allowed for the visualization of the distribution lines of non-metallic inclusions and the flow patterns of the particles. The obtained observations provided essential insights into the dynamics of adhesion formation and were instrumental in supporting the proposed theoretical model.

The mechanism of adhesion formation is described in terms of the conditions that favor changes in apparent viscosity, ultimately leading to plasticity and fluidity of the material. These transformations enable the development of the accretions observed in the collected samples. A schematic representation of the proposed mechanism is presented in Figure 1, which illustrates the sequence of events leading to the accumulation of material on the internal surfaces of the particle separator.

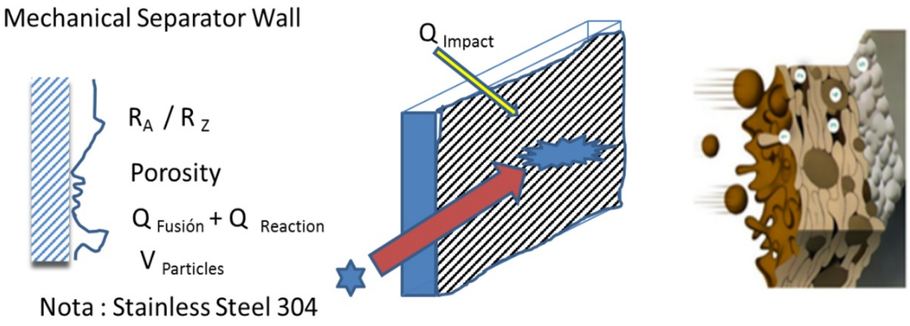

Fig. 1. (a) Represents the pattern of impact of a particle against a metal wall (b) Indicates how the layers of the crust are formed by thermos-adherence.

In this mechanism, the solid particles heat up during the conversion reaction of ferrous iron to metallic iron. When these particles impact the steel surface and interact with its roughness, they release sufficient energy (Fig. 1a), causing them to become viscous. This viscous material:

1. Crawls along the surface driven by the gas flow,
2. Superimposes over previous surface imperfections,
3. Creates pores and generates fayalite-type silicates (Fig. 1b).

By analogy, this is similar to a surface thermo-adhesion process.

A. Energy Balance

The energy balance, previously calculated and published in [4], is summarized in Table 1.

Tabla 1. Energy balance within the particle separator

Symbol	**Meaning**	**Unit (kJ/mol)**	**Unit (kcal/mol)**
E_c	Energy per impact	103	30
E_v	Energy per vacancy	474	113
E_p	Energy per particle collision	13	3
E_i	Energy due to inelastic collision	70	17
Total	**Total energy**	**660**	**163**

The calculated total energy of 660 kJ/mol agrees with the reported energies for the formation of fayalite [5], as well as with the softening point of silica, approximately 599 kJ/mol at 2000 °C [6]. This confirms that the formation of fayalite within the studied temperature range is thermodynamically possible under intermediate viscous conditions.

B. Effect of Temperature on Apparent Viscosity

The total energy calculated is 61 kJ/mol greater than the 599 kJ/mol required for the softening of SiO_2. This matches the range of 61–65 kJ/mol, considered as the compensated activation energy required for silicate particles to undergo deformation and creep.

The particles in the gas-solid mixture not only possess appropriate thermal conditions but also contain iron particles undergoing a transient phase change from ferrous oxide (FeO) to metallic iron. This transformation releases additional energy associated with vacancies.

When silica particles collide with wustite particles, they behave similarly to thermally coated particles projected at high velocity onto a metal surface. Under these conditions, fusion reactions between the particles are inevitable, ensuring the formation of the viscous phase required for adhesion.

C. Adhesions of Silicate by Action of Fluence

The formation of a viscous FeO-SiO_2 phase, with minor amounts of MgO and metallic iron, occurs under a gas velocity of approximately 75 m/s at the cyclone entry. This creates a deformation force that induces thermo-viscous behavior, combined with creep.

Considering creep behavior requires knowledge of the melting temperature (T_m) of metallic iron. The studied temperatures ranged between 620 °C and 700 °C, which correspond to 0.3–0.5 T_m. In this range, creep behavior depends logarithmically on temperature and follows an Arrhenius-type relationship.

Using the slope-change method, activation energy for creep was calculated:

$$E_a = 65 \text{ kcal/mol}$$

When plotting the logarithm of deformation rate against the inverse of absolute temperature, a linear relationship was obtained, resulting in:

$$E_a = 62 \text{ kcal/mol}$$

These values align with those reported for self-diffusion in this temperature range [7].

D. Relationship Between Thermoplasticity and Creep

Since the activation energy for creep matches that of self-diffusion, the creep mechanism is controlled by mixed diffusion. This involves the non-conservative motion of dislocations, including edge dislocations and screw dislocations.

At the studied strain rates and temperatures, vacancy concentration remains near equilibrium, with only minor perturbations.

Using data from [8], vacancy superheights were estimated for the range 150 °C–700 °C (0.23 T_m–0.54 T_m) at various creep rates. From thermodynamic considerations, the energy released by defined particle sizes in the separator was quantified.

Starting from Cauchy's first law and multiplying both sides by viscosity (v):

$$\rho \frac{D}{Dt}\left(\frac{v^2}{2}\right) = (\nabla \cdot T)v + fv \tag{1}$$

If the body force is the gradient of a potential, $f = -\nabla\phi$, then:

$$\rho \frac{D}{Dt}\left(\frac{v^2}{2}\right) = (\nabla \cdot T)v + \rho\frac{D\phi}{Dt} \tag{2}$$

Rearranging terms:

$$\frac{D}{Dt}\left(\frac{v^2}{2} + \phi\right) = (\nabla \cdot T) \cdot v + T : \nabla v \tag{3}$$

Here, the product $T : \nabla v$ represents viscous dissipation (E_V).

The formation of cylindrical accretions above 750 °C and pressures over 10 bars is feasible when the gas molecular weight is near 10 g/mol. These conditions validate that particle collisions release enough energy to initiate thermoviscosity and soften the particles.

E. Effect of Temperature on Ferrous Transformations

Once the apparent viscosity and creep participation were quantified, the next step was to analyze the critical temperature for thermoplasticity onset. Partially reduced iron oxide particles heat up through gas flow, creating a precondition linked to sticking tendency.

This tendency varies by mineral type and composition. Figure 2 illustrates the transition from wustite to metallic iron, highlighting the effect of reduction rate on critical adhesion temperature.

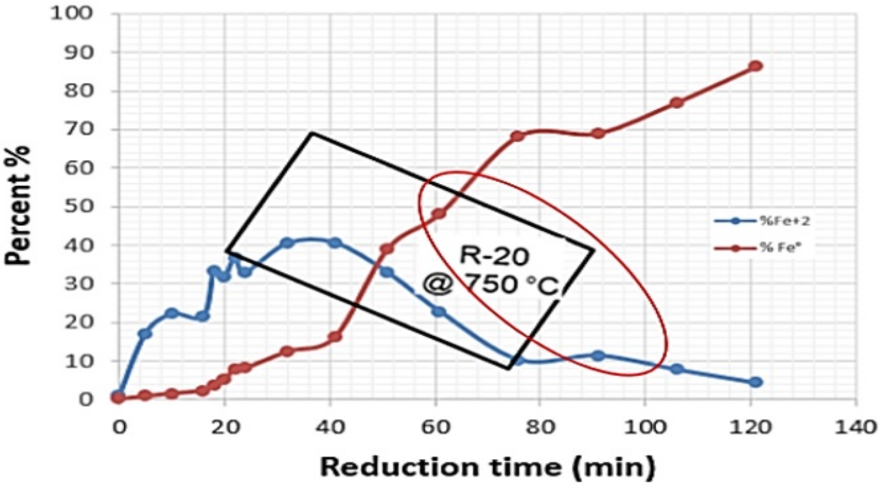

Fig. 2. Transformation of ferrous iron into metallic iron in the studied area [9]. .

Depending on the reduction stage, hot particles exhibit varying sticking potentials, directly impacting adhesion formation within the fluidized bed reactor.

The data used to develop Figure 3 were obtained from industrial tests conducted in a C.F.D. plant using mixed Venezuelan and Australian mineral mixtures, as well as tests with 100% Australian mineral. From this figure, it is possible to estimate the ranges of temperature values at which sticking of iron particles occurs. These particles, partially metallized, exhibit adhesion behavior as a function of the formation of metallic iron within the particle separators during their transfer into these systems. The temperatures obtained can be assumed to represent actual working or operating conditions.

This temperature creates an activated state in the particles, predisposing them to different stages of the sticking phenomenon (Figure 3), such as bogging or particle agglomeration in the fluidized bed, crustal adherences in the upper cylindrical section of the particle separators, and sintering in the returning particles located in the lower portion of the separator leg.

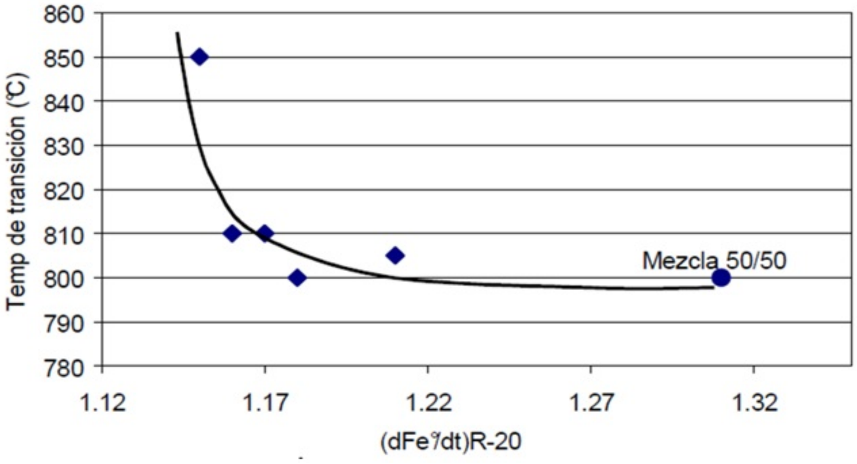

Fig. 3. Effect of the metallization rate on the critical adhesion temperature [9].

This activated adhesion potential, developed by the partially reduced iron oxide particle, enters the particle separator along a trajectory that eventually results in collisions with the clean steel surface. These collisions occur during the initial accretion formation or subsequent accretion development.

The kinetic behavior of this process can be mathematically expressed by Equation (4):

$$T_{\text{rop}} = 3987\left(\frac{d\text{Fe}^0}{dt}\right)^2 - 9974\left(\frac{d\text{Fe}^0}{dt}\right) + 7027 \tag{4}$$

where:

- T_{rop} = Actual operating temperature (°C)
- Fe^0 = Metallic iron content
- t = Time

The correlation factor R^2 obtained for this model was 0.7684, representing a reliability of industrial data greater than 85%, which is considered very acceptable given the inherent variability of industrial processes.

III. METHODOLOGY

At this point, the appropriate theoretical and thermodynamic basis for the proposed mechanism has been clearly established, as well as the empirical relationships between the critical adhesion temperature, viscosity, and creep variables of the silicates formed during solid-particle collisions, as well as the impact between the particles and the particle separator surfaces. Each of the steps taken to obtain the results presented in the following section is explained below.

In the theoretical foundation of the thermos-adhesion phenomenon, this article establishes a solid foundation of thermodynamic principles, viscosity physics, and mechanics of silicate behavior, focusing on the formation of thermal adhesions in ceramic materials. The central hypothesis relates the relative viscosity and plasticity of the system to the activation energy required for adhesion formation.

Sampling involved taking material samples adhering to the metal surface, specifically in the cylindrical area of the mechanical separator in the wustitic reactor. These samples underwent metallographic analysis and a Bauman test to determine the stratification of the adhering layers. Additionally, chemical composition analyses were performed to determine the initial conditions and relevant physical properties of the samples.

In the operating conditions analysis, operational data were collected from production processes, including temperature ranges, pressure, gas composition, working pressures, and material reduction percentage. The impact of flow rate and thermal distribution within the reactor was also considered to better understand the dynamic behavior of the system.

The process modeling was carried out using an Excel spreadsheet to simulate flow behavior and the interaction between particles and surfaces in the mechanical separators. This modeling made it possible to identify the areas of greatest adhesion and evaluate the influence of variables such as temperature, viscosity, and collision energy. The calculation of collision and adhesion energies was summarized in a table, quantifying the contribution of each type of energy and the conditions under which adhesion formation occurs, based on the theory of particle collisions and an adaptation of the McClean adhesion equation.

The viscosity and plasticity analysis discussed in this study establishes a direct relationship between the viscosity of the system and the formation of accumulations as a function of temperature and chemical composition, applying rheological models specifically developed for ceramic and silicate materials.

IV. RESULTS

A. Sample Preparation

To corroborate the observed system, solid industrial samples were taken from the accretions formed inside the particle separators. As shown in Figure 4, these samples were cut, roughened, and etched with a 12% sulfuric acid solution to reveal the flow lines of the formations of the different layers in the opposite direction of the gas flow entering the cyclone separator.

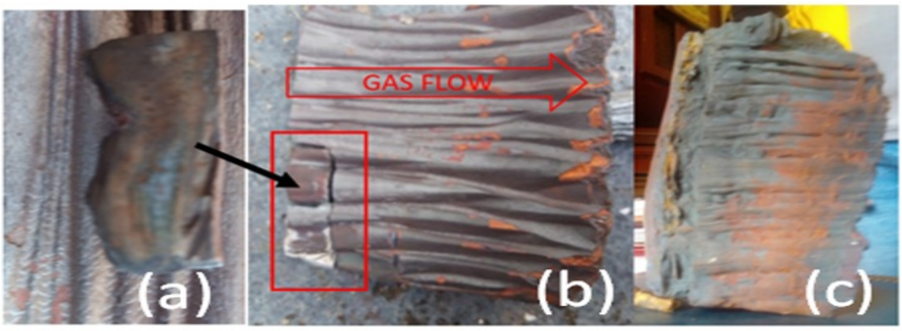

Fig. 4. Sample of gas flow accretion and creep lines. (a) Flow lines and layers of deposited material attacked with 12% sulfuric acid. (b) Accelerations formed by the collision between particles. (c) Flow lines of the adhesions in the metallic crusts, at the point where the impact speed decreases.

This proposed mechanism meets both the thermodynamic and process conditions for accumulations to occur.

B. Proposed coefficient for iron oxide dust

The materials, depending on the temperature range, have a *thermos-adherence constant* [9] as follows: 0–0.3 T_M, 0.3–0.5 T_M, 0.5–0.9 T_M and 0.9–1.0 T_M. Here, T_M represents the melting temperature of pure iron, which is 1534 °C. Based on this information, the following results were obtained.

Tabla 2. Comparison of obtained calculation results

Temperature (°C)	K Thermo Creep	Temp. Creep (°C)
461	0.3	460
700	0.5	767
1400	0.9	1381

C. Influence of temperature on the thermal adherence constant

The value of the coefficient of adhesion in the temperature range between 600 °C and 700 °C oscillates between 0.478 and 0.514, with a correlation of 0.977, for ceramic materials (Fig. 5). These values align with those proposed by McLean for metals in a diffusion-controlled creep process, simulating similar flow conditions to those of liquid metals.

This relationship assumes a combined effect of working pressure on temperature, which in turn affects the particles' velocity, gas molecular weight, and oxide properties, resulting in a plastic activated stage responsible for the sticking tendency.

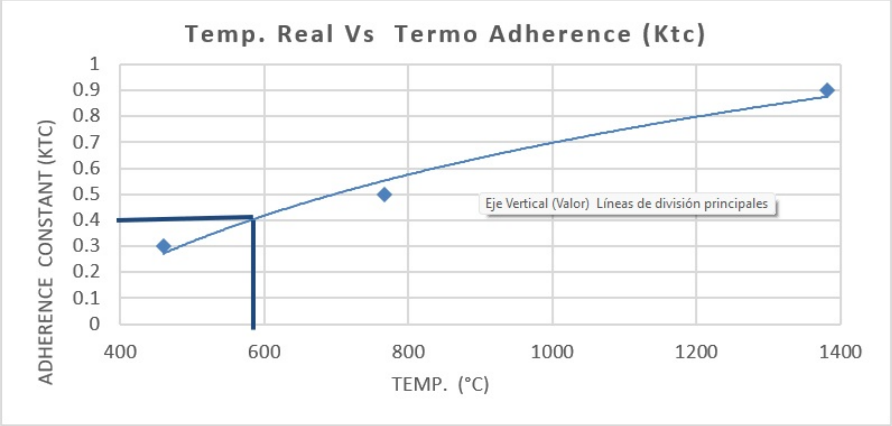

Fig. 5. Result of the thermos-adherence constant.

$$K_{Tc} = 0.5491 \cdot \ln(Tr) - 3.095 \tag{6}$$

Where:

- K_{Tc} = Adherence constant (dimensionless)
- Tr = Actual operating temperature (°C)

Using the temperature difference for the occurrence of thermoplasticity, in the range of 600 °C up to the melting point of the newly formed iron, the value of K_{Tc} obtained was 0.478. This represents a modification of the McLean equation applied to ceramic systems.

The modified equation to define the appearance of yield stress in the studied system is represented as:

$$Tc = 2.09121 \cdot Tr - 10^{-12} \tag{7}$$

Where:

- Tc = Thermal adherence constant
- Tr = Actual working temperature (°C)

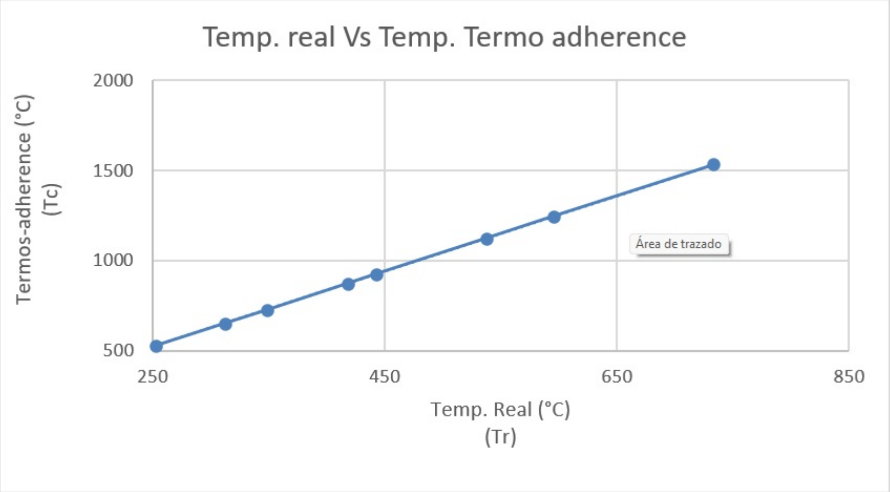

Fig. 6. Formation of thermal adhesion as a function of real operating temperatures.

D. Energy Activation in the Study System

The formation of adhesions as shells in the cylindrical zone, under the studied temperature and pressure conditions, is feasible in fluidized bed processes when the gas molecular weight is 10 g/mol. A temperature gradient of 30 °C between the hot gases and solid particles was assumed.

The Arrhenius equation used for modeling the activation energy is given by:

$$\log K = \log A - \frac{E_A}{R} \cdot \frac{1}{T} \tag{8}$$

Where:

- K = Reaction rate constant
- A = Molecular collision frequency
- E_A = Activation energy (kcal/mol)
- R = Ideal gas constant

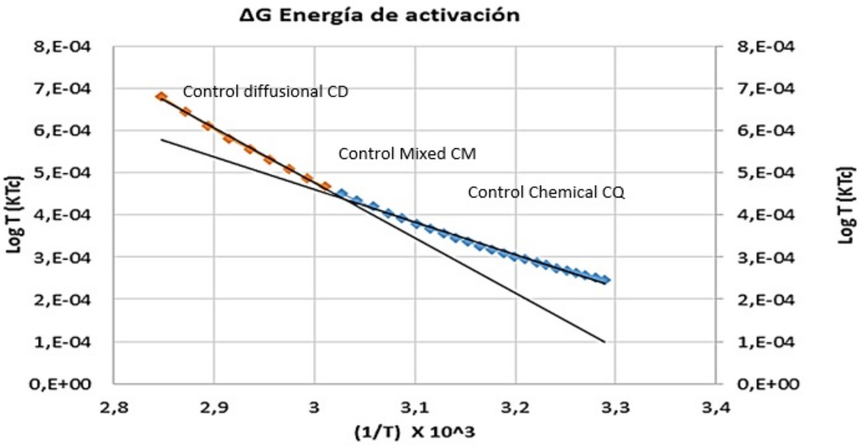

Fig. 7. Activation energy through the Arrhenius relationship. Diffusional Control (CD); Chemical Control (CQ); Mixed Control (CM).

- T = Absolute temperature (K)

The activation energy values help identify the predominant mechanism:

1. *Diffusional Control*

$$\log k = -0.013 \cdot \frac{1}{T} + 0.0045 \tag{9}$$

2. *Mixed Control*

$$\log k = -0.052 \cdot \frac{1}{T} + 0.0038 \tag{10}$$

3. *Chemical Control*

$$\log k = -0.0008 \cdot \frac{1}{T} + 0.0028 \tag{11}$$

E. Interpretation of Results

The temperature's influence follows particle collision theory as described by Arrhenius.

- Chemical control:Leads to bogging and particle accumulation in the bed.
- Diffusion control: Results in sintering phenomena.
- Mixed control: Affected by changes in temperature and thermal adherence constant.

The calculated activation energies are:

- Diffusional control: 5.949 kcal/mol (slope = 1.3×10^3)
- Chemical control: 3.661 kcal/mol (slope = 0.8×10^3)
- Mixed control: 4.805 kcal/mol (slope = 1.05×10^3)

These values confirm the transition between different control mechanisms depending on the operating temperature and system conditions.

CONCLUSIONS

Analysis using particle collision theory reveals the energies required for adhesion in separators within the temperature range of 600–700 °C. This process is fundamental for the formation of fayalite in the presence of ferrous oxide, silica, and magnesium oxide.

The adhesion coefficients for ceramic systems such as the one studied, between 600 °C and 700 °C, range from 0.478 to 0.514, with a correlation of 0.977. These values differ from those proposed by McClean for metals but are consistent with the expected range and are applicable to a diffusion-controlled creep process, analogous to the flow of liquid metals in thermal spraying.

Based on this theoretical principle, a yield point constant was determined, which modifies McLean's theory for ceramic metals. This modification is justified by the energy released in the particle separator, which, upon exceeding the transformation point from solid to liquid oxides, induces thermal adhesion and plasticity in the oxides.

The results indicate that the energy released in the cyclone separator (660 KJ/mol) is significantly higher than the fusion energy of silicon oxide at 2000 °C (599 KJ/mol), with an excess of 61 KJ/mol.

Finally, the application of the Arrhenius equation allowed the determination of the activation energies for the chemical (CQ), diffusional (CD), and mixed (CM) controls, resulting in values of 5.94 kcal/mol (CD), 4.81 kcal/mol (CM), and 3.66 kcal/mol (CQ), respectively. The total activation energy for fayalite formation was found to be 14.41 kcal/mol, a value very close to the theoretical prediction.

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