

## The Improved Low Temperature Digestion (ILTD) Process and its Recent Developments

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### Abstract

The Improved Low Temperature Digestion (ILTD) Process is flexible and offers many benefits, compared with the Conventional Low Temperature Digestion Process, including the following:

- material and energy cost reduction of 15-40 %;
- savings in the chemical caustic losses by 30-80 %, depending on the process configuration and conditions;
- high precipitation liquor productivity of at least 90 g/L;
- higher production rate in the digestion by up to about 25 %;
- reduced quantity of bauxite residue generation by up to 30 % and with an extremely low soda and possibly high iron content;
- recovery of combined soda and alumina from desilication product (DSP);
- extending the options for the industrial utilization of the bauxite residue (processing of bauxite possibly with no waste to dispose of).

The ILTD Process was originally developed for processing gibbsitic type bauxites, which comprise nearly 90 % of the World's bauxite reserves. This paper reviews the current status of the development, the options for the utilization of the bauxite residue having low soda and possibly high iron content and that for the desilication by-product. A recent Case Study revealed that the ILTD Process is viable for processing high silica gibbsitic bauxites/laterites which contain e.g. 5.5 % or more of R.SiO<sub>2</sub> (Reactive silica). The breakthrough results (savings in the material and energy costs of about USD 60-75/t of alumina) greatly extend the viable processing of low-quality bauxites, even aluminous laterites that have been considered so far to be sub-economic. The worldwide amount of sub-economic bauxite resources of 10-25 Gt is deemed to be a conservative estimate. The updated ILTD Process calls for a shift of paradigm of the Bayer process since the alumina in kaolinite in bauxite can be extracted in a viable way.

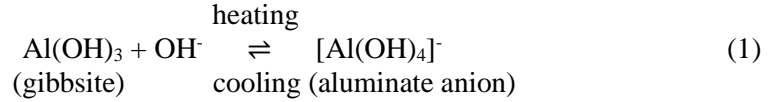
**Keywords:** Production costs, Bauxite residue (red mud), Bayer process, ILTD Process, Waste free process.

### 1. Introduction

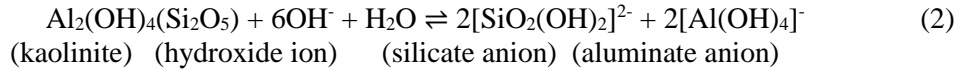
The two substantial unit operations of the Bayer process are the dissolution of the hydrated alumina minerals (digestion), and the crystallization of Al(OH)<sub>3</sub> (precipitation). The digestion process step highly depends on the mineralogical composition of the bauxite. The two major forms of digestion are: the low temperature digestion which was developed for the gibbsitic type bauxites and is operated at about 140-150 °C; and the high temperature digestion for the boehmitic and diasporic bauxites, applied at about 240-270 °C. Different flowsheets have been developed for the processing of bauxites with mixed mineralogy such as double digestion for mixed gibbsitic-boehmitic bauxites. It is worthwhile to mention the sweetening process as an occasional embodiment of the Bayer process and the serial combined and the parallel combined Bayer-sintering processes. The review of these non-conventional flowsheets is beyond the scope of the present paper.

## 2. Dissolution of Gibbsite, Transformation of Kaolinite into DSP

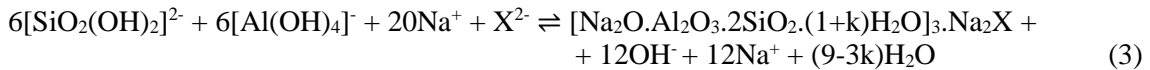
The reaction equation with the arrow to the right symbolizes the dissolution of gibbsite (digestion), with the arrow to the left depicts the precipitation of Al(OH)<sub>3</sub>.



Dissolution reaction of kaolinite



The sodium aluminium hydrosilicate (desilication product, DSP, mostly sodalite) compound that forms in the liquid phase as per the desilication reaction equation (3) at temperatures of about 140-160 °C is described in a novel form [1]:

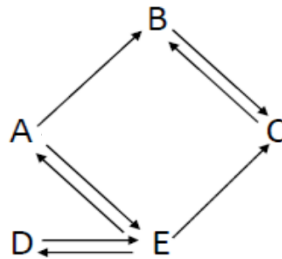


where X: 2OH<sup>-</sup>, CO<sub>3</sub><sup>2-</sup>, SO<sub>4</sub><sup>2-</sup>, 2Cl<sup>-</sup>, 2[Al(OH)<sub>4</sub>]<sup>-</sup>, etc., k = 0-1

## 3. The Reaction Mechanism and Rate Equations of the Dissolution of Gibbsite and Transformation of Kaolinite to DSP

### 3.1 The Reaction Mechanism

The kinetics of the dissolution of gibbsite and transformation of kaolinite to DSP was tested at about 100 °C and under a wide range of caustic concentrations [2]. The mathematical modeling of the set of kinetic tests enabled the comprehensive reaction mechanism to be determined [3], that is depicted with the following schematics [4].



**Figure 1. Reaction mechanism of dissolution of gibbsite and transformation of kaolinite to DSP.**

**A: kaolinite, B: dissolved silica, C: sodium aluminium hydrosilicate (DSP), D: gibbsite, E: dissolved alumina**

### 3.2 Reaction Rate Equations of Dissolution of Gibbsite and Kaolinite, Formation of Desilication Product

The reaction rate equations, which best describe the substantial reactions of the dissolution of gibbsite and the transformation of kaolinite into desilication product are shown below (4)-(6). The rate equation for the dissolution of kaolinite that was developed originally for the pre-desilication [3] has been adopted to the conditions of the low temperature digestion [1].

$$r_1 = dc_A/dt = k_1 \mathbf{AH} c_A \text{ dissolution of kaolinite} \quad (4)$$

$$r_2 = dc_B/dt = k_2 (c_B - c_B^{eq}) \text{ formation of DSP} \quad (5)$$

$$r_3 = dc_D/dt = k_3 \mathbf{AH} c_D \text{ dissolution of gibbsite} \quad (6)$$

$$\mathbf{AH} = 2 \times 17 [(c_E^{eq} - c_E)/102 - c_B/60] \text{ reactive OH}^- \text{ concentration} \quad (7)$$

where  $c_A, \dots, c_E$  are the concentrations of the constituents as denoted in the schematic of the reaction mechanism (Fig. 1),  $\mathbf{AH}$  is the reactive  $\text{OH}^-$  concentration, 17, 102 and 60 are the molar masses of  $\text{OH}$ ,  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$ , respectively.  $c_B^{eq}$  and  $c_E^{eq}$  are the equilibrium solubilities for dissolved silica and alumina, respectively.  $k_1$ ,  $k_2$  and  $k_3$  are the kinetic rate constants, to be determined experimentally.

#### 4. Issues with the Pre-desilication and the Conventional Low Temperature Digestion

Investigations of the pre-desilication and the low temperature digestion and experiences of plant operations revealed that there are issues with both unit operations, as listed below.

##### 4.1 Issues with the Pre-desilication

- Investment and working capital are required
- Part of the gibbsite may transform to (pseudo-)boehmite during pre-desilication (some 4 rel.% in 6 hours at 103 °C was measured for a Fria bauxite, Guinea)
- DSP formed at about 100 °C partially dissolves during the Conventional Low Temperature Digestion (CLTD)
- Different types of DSP form at different temperatures and caustic concentrations.

##### 4.2 Issues with the Conventional Low Temperature Digestion

- The conditions of the digester reactor are designed for the nearly complete crystallization of the dissolved reactive silica as DSP (retention time about 30-60 minutes) and **not** for the complete dissolution of gibbsite which requires about 3-5 minutes
- The dissolved silica content in the digestion effluent can not be maintained very close to the equilibrium, therefore excessive silica scalings occur in the test tank (digestion) liquor preheaters
- The hydrate product is contaminated with silica
- A part of the digestion capacity of the Test Tank Liquor is not utilized in order to prevent excessive formation of (pseudo-)boehmite due to a side reaction of the dissolution of gibbsite
- The A/C ratio ( $\text{Al}_2\text{O}_3/\text{C}$ , where C is caustic soda concentration as  $\text{Na}_2\text{CO}_3$ ) in the liquor for precipitation is restricted, thereby the precipitation liquor productivity is less than it could be
- Significant alumina losses occur (can be about 5-7 %, or even more, if both pre-desilication and LT (low temperature) digestion are applied) due to the hydrothermal formation of (pseudo-)boehmite from the gibbsite.

#### 5. Kinetic Tests of Dissolution of Gibbsite and Transformation of Kaolinite under the Conditions of the ILTD Process

In the course of bench scale kinetic tests [4] the following features were applied:

- Trombetas bauxite concentrate from Brazil was used as feedstock. Its chemical composition is in Table 1 (mineralogical composition: 51.8 %  $\text{Al}_2\text{O}_3$  in gibbsite, 0.8 % in boehmite and 2.2 % in kaolinite, 2.6 %  $\text{SiO}_2$  in kaolinite and 0.5 % as quartz).
- Physical modeling of the two-stream digestion was applied (the preset digestion temperature was reached within 1-2 minutes)

- Repeated digestion kinetic tests were applied
- The digestion temperatures were set to 130 °C and 150 °C
- The agitation was fairly vigorous (rpm of 200/min and 400/min).

Process parameters of the kinetic testing at the ILTD conditions were:

- Test Tank Liquor caustic concentration - 269 g/L C, A/C ratio - 0.325
- Digestion temperature - 150 °C
- Agitation - 200 rpm
- Reactive OH<sup>-</sup> concentration < 3 g/L, (target A/C ratio ≥ 0.82).

The results of the kinetic tests at the ILTD digestion are shown in Figures 2-6. The points represent the retention times when the slurry of the reactor was sampled. After this the solid phase was separated from the liquid and both phases were analyzed. The arrows with the caption “BR separation” at 6 minutes reaction time relate to the proposed condition of the ILTD Process.

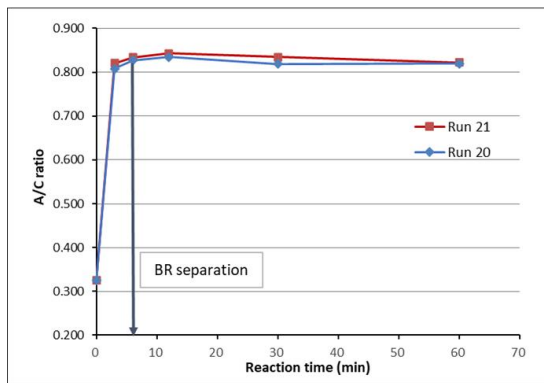


Fig. 2. A/C ratio vs reaction time.

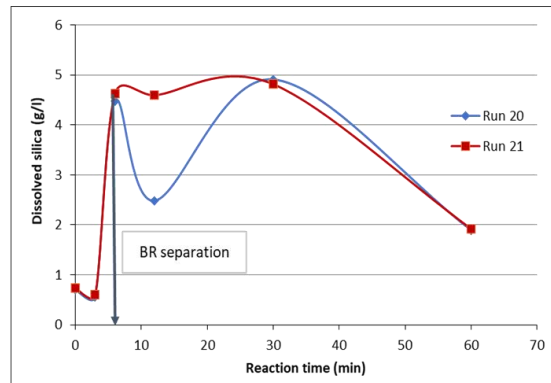


Fig. 3. SiO<sub>2</sub> concentration vs reaction time.

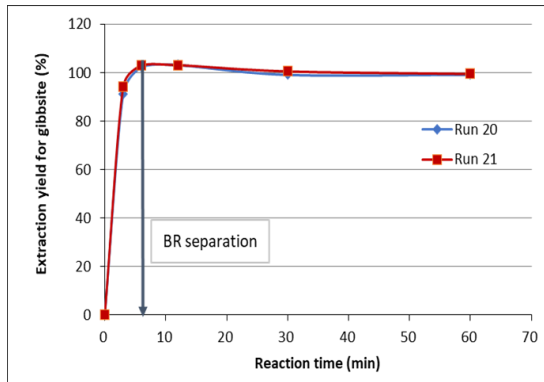


Fig. 4. Al<sub>2</sub>O<sub>3</sub> extraction yield for gibbsite.

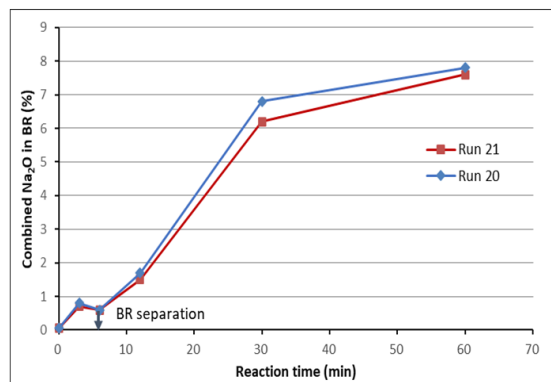


Fig. 5. Na<sub>2</sub>O content in bauxite residue.

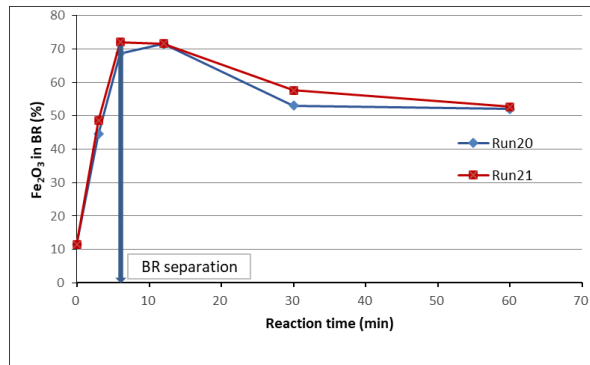


Fig. 6. Fe<sub>2</sub>O<sub>3</sub> content in BR vs reaction time.

The compositions of the bauxite feedstock, the bauxite residues (BR) of the Conventional LT Digestion and the ILTD Processes derived from the bench scale kinetic tests are shown in Table 1. The bauxite and the BRs were analyzed with wet chemistry where ICP and AAS methods were included.

**Table 1. Chemical compositions of Trombetas bauxite (Brazil), bauxite residues (BR) of the Conventional and Improved Low Temperature Digestions [4].**

Composition, %	Trombetas bauxite	BR of CLTD Process	BR of ILTD Process
Al <sub>2</sub> O <sub>3</sub>	54.65	14.3	9.2
SiO <sub>2</sub>	3.13	13.2	5.8
Fe <sub>2</sub> O <sub>3</sub>	11.51	52.7	70.4
(Fe)	8.05	36.9	49.2
TiO <sub>2</sub>	0.82	3.7	4.1
Na <sub>2</sub> O <sub>combined</sub>	-	8.0	0.6
Na <sub>2</sub> O <sub>comb</sub> /SiO <sub>2</sub>	-	0.6	0.1
LOI	29.53	7.1	6.1

**Remarks:**

BR of CLTD: average of tests No 17 and 18, 150 °C at 60 min, **AH > 11g/L**

BR of ILTD: average of tests No 20 and 21, 150 °C at 6 min, **AH < 2g/L**

## 6. Fundamental Considerations and Principal Features of the ILTD Process

### 6.1 Fundamental Considerations

A fundamental consideration of the ILTD Process is that the dissolution reactions of gibbsite and kaolinite are best explained as a competition of gibbsite and kaolinite for the reactive OH<sup>-</sup> ions. Gibbsite is present in a significantly larger amount (about 10-25 times higher) and, as kinetic studies revealed, has a higher dissolution reaction rate than that of kaolinite (about 10 times higher) during the Low Temperature Digestion. Therefore at low reactive OH<sup>-</sup> target concentrations (< 6 g/L, preferably < 3 g/L), gibbsite consumes most of the common reactant, the reactive OH<sup>-</sup> ions, before kaolinite totally dissolves.

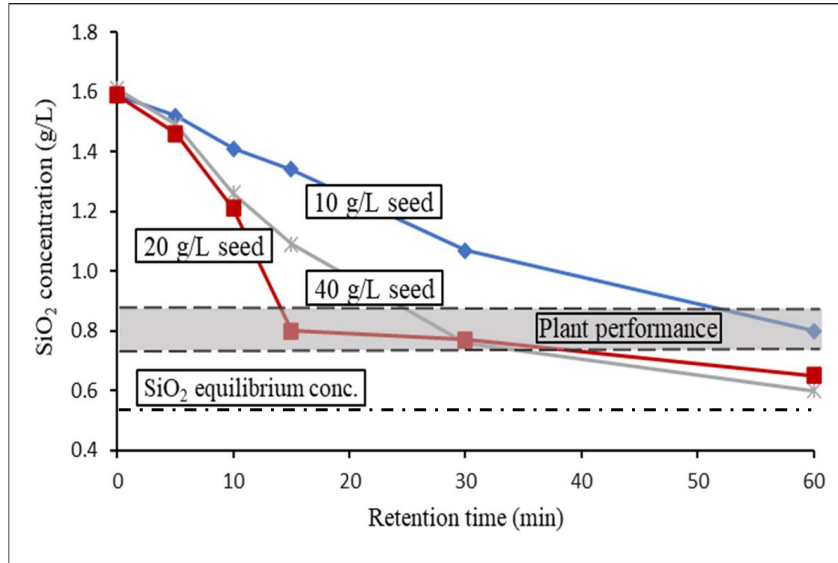
### 6.2 Principal Features of the Improved Low Temperature Digestion (ILTD) Process

The ILTD Process conditions are as follows:

- no pre-desilication is needed
- digestion temperature about 150 °C
- high A/C ratio, A/C ≥ 0.800 (**AH** < 6 g/l), short digestion retention time, about 3-5 mins
- separation of the low soda bauxite residue just after the digestion, preferably with pressure decanter and/or Hi-Bar Filtration, at about the digestion temperature
- seeded pressure crystallization of the Desilication Product (DSP) out of the liquid phase.

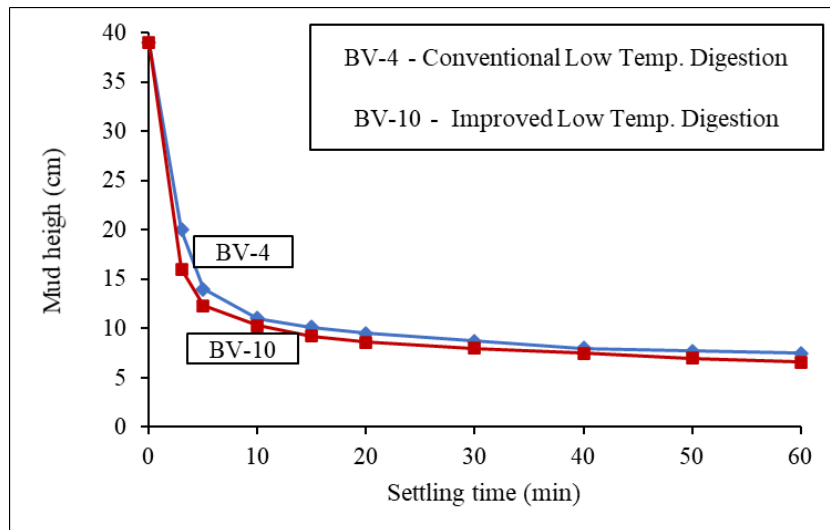
## 7. Seeded Pressure Liquor Desilication and Comparative Settling Tests [5]

Bauxite from Pijiguaos mine (Venezuela) with low reactive silica content was digested at 140 °C for 5 minutes, and the pregnant liquor obtained after the separation of the BR (C - 217 g/L, A/C ratio - 0.729) was submitted to seeded pressure liquor desilication at 150 °C, at different DSP seed dosages of 10, 20, 40 g/L.



**Figure 7. SiO<sub>2</sub> concentrations vs retention time of seeded pressure liquor desilication at 150 °C.**

The results of a comparative settling test are shown in Figure 8. Residues of Pijiguaos bauxite were settled in settling tubes. BV-4 stands for the Conventional and BV-10 for the Improved Low Temperature Digestion. The digestion conditions for the CLTD were 140 °C, 75 min, A/C ratio - 0.723, for the settling C - 221 g/L, A/C ratio - 0.687, settling additives Nalco - 7879 60 g/t, flour of 2 kg/t were applied. For the ILTD the digestion conditions were 140 °C, 5 min, A/C ratio - 0.782, for the settling C - 221 g/L, A/C ratio - 0.697, settling additives Nalco - 7879 60 g/t, flour 2 kg/t were used. The separation characteristics might be different for different residues; therefore, they should be tested for each feedstock.



**Figure 8. Comparative settling tests for the CLTD and the ILTD.**

### 8. Block Flow Diagram of the ILTD Process

The block flow diagram of the ILTD Process is shown in Figure 9. The blocks highlighted in yellow relate to unit operations which are different from the Conventional LTD Process [4].

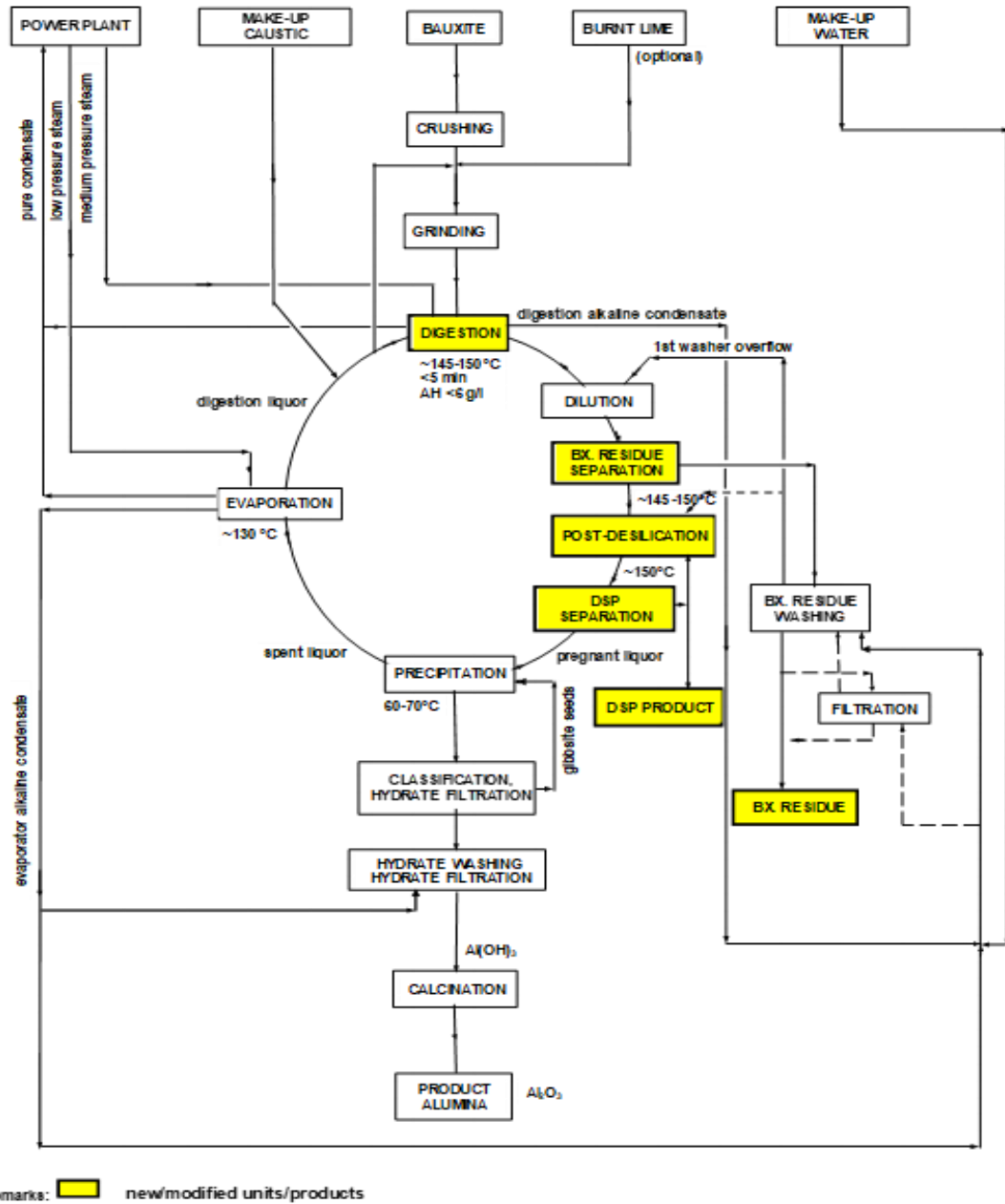


Figure 9. Block flow diagram of the Improved Low Temperature Digestion (ILTD) Process.

**9. Comparison of the Principal Process Parameters of the Conventional Low Temperature Digestion and an ILTD Processes**

**Table 2. The principal process parameters of a Conventional and an ILTD Process.**

	<b>CLTD Process</b>	<b>ILTD Process</b>
Pre-desilication	used in several plants	not used
Digestion (Test Tank) liquor C, g/L	291	312
Digestion (Test Tank) liquor A/C ratio, -	0.41	0.41
Digestion (Test tank) liquor, m <sup>3</sup> /t alumina	10.88	8.58
Digestion temperature, °C	145	150
Holding time, min	≈60	< 5
Digestion A/C ratio, -	0.76	0.82
Reactive OH <sup>-</sup> conc., g/L	9.4	4.0
Digestion yield for gibbsite, %	≈96.5	99.25
Reacted kaolinite, %	100	≈60
SiO <sub>2</sub> in the reactor effluent, g/L	≈0.8-1	≈4.5
Bauxite residue separation temperature, °C	100	(145-)150
Post-desilication temperature, °C	-	(145-)150
DSP seed dosage, g/L	-	≈30
Post-desilication holding time, min	-	30-60
SiO <sub>2</sub> conc. after post-desilication, g/L	-	≈0.6
Pregnant liquor C, g/L	265	265
Pregnant liquor A/C ratio, -	0.75	≥ 0.78
Pregnant liquor SiO <sub>2</sub> concentration, g/L	≈0.8-1.0	≈0.7
Liquor causticity, %	98	98
Precipitation liquor productivity, kg/m <sup>3</sup>	86.5	≥ 94.3

Table 2 relates to a Case Study in an alumina refinery in Latin America, where Trombetas bauxite with very low organic content was the principal feedstock. The liquor causticity was extremely high (98 %.). Therefore, the liquor productivity at the precipitation for the CLTD was significantly higher than in most of the other refineries in the region, and consequently for the ILTD Process very high productivity (≥ 94.3 kg Al<sub>2</sub>O<sub>3</sub>/m<sup>3</sup> pregnant liquor) is anticipated.

**10. Novel Developments, Cost-Benefit Analysis, Status of the ILTD Process**

**10.1 Recent Process and Economic Calculations, the Updated ILTD Process**

There are very significant amounts of bauxite resources, which, at the conventional low temperature digestion conditions proved to be sub-economical due to their high reactive silica (R.SiO<sub>2</sub>) content. Therefore, they were correctly left out when the reserves were estimated.

The theoretical chemical composition of DSP from the ILTD Process is close to nepheline in composition. The crystal water and CO<sub>2</sub> of DSP were left out for the comparison (Table 3). Therefore, some unit operations of the processing of nepheline (similarly to other sintering

processes) can be well utilized in the ILTD Process. The seeded pressure desilication of the pregnant liquor is an example. Some 90 % of the soda and alumina content of the DSP by-product of the ILTD Process can be recovered by a small sintering-leaching unit. The dicalcium-silicate (belite, C<sub>2</sub>S) by-product of sintering-leaching of DSP is welcome by the cement industry. The ILTD Process completed with soda and alumina recovery from the DSP by-product is called **updated ILTD Process**.

**Table 3. Composition of nepheline and CO<sub>3</sub>-sodalite.**

	<b>Nepheline</b>	<b>CO<sub>3</sub>-sodalite</b>
SiO <sub>2</sub> , %	43.1	39.4
Al <sub>2</sub> O <sub>3</sub> , %	36.6	33.5
Na <sub>2</sub> O, %	20.4	27.1
LOI, %	-	-

**Remarks:**

- K<sub>2</sub>O content in Nepheline ((Na<sub>0.75</sub>,K<sub>0.25</sub>)<sub>2</sub>O.Al<sub>2</sub>O<sub>3</sub>.2SiO<sub>2</sub>) converted to Na<sub>2</sub>O
- CO<sub>3</sub>-sodalite (3(Na<sub>2</sub>O.Al<sub>2</sub>O<sub>3</sub>.2SiO<sub>2</sub>.2H<sub>2</sub>O).Na<sub>2</sub>CO<sub>3</sub>) without CO<sub>2</sub> and H<sub>2</sub>O content

Another novel development of the updated ILTD Process has been its application for processing of low-quality bauxites and aluminous laterite. India has large stocks of this kind of materials, and they have been considered sub-economical [6]. Some of them have been used as a material for blending with high quality imported bauxites, possibly after some mechanical beneficiation [7].

Tentative process and economic calculations for a typical Eastern Ghats aluminous laterite have been carried out for an Indian East Coast Refinery starving for indigenous bauxite. For the composition of the aluminous laterite, the presentation of Nandi and Mahadevan is referred [8]. The results of the calculations are shown in Table 4. As it has been demonstrated, Material and Energy cost savings of nearly 60-75 USD/t of alumina could be achieved if the ILTD Process was implemented and applied instead of the CLTD Process. It might be a paradox that the updated ILTD Process can be more viable than the CLTD Process. Despite good grade bauxite reserves being available on the East Coast, by using the ILTD Process not only the low-grade resources can be utilized, but the resource pool can be increased geometrically, thus enabling an increase in availability for both indigenous refineries and also cater for the export market.

**10.2 A Tentative Cost-Benefit Analysis**

The process costs and benefits highly depend on the location of the bauxite/laterite resources, their quality, the location of the alumina refinery, the unit prices and several other factors. A tentative cost-benefit assessment has been carried out for an Indian alumina refinery, using the calculated material and energy costs and savings as shown in Table 4. The Return of Investment (ROI) was determined to be between 1 and 2 years, the most probable values are 1.3 and 1.7 years, depending on whether the soda and alumina contents of DSP are recovered within the refinery by sintering or not. No revenues from selling of the ILTD Process to third parties have been considered.



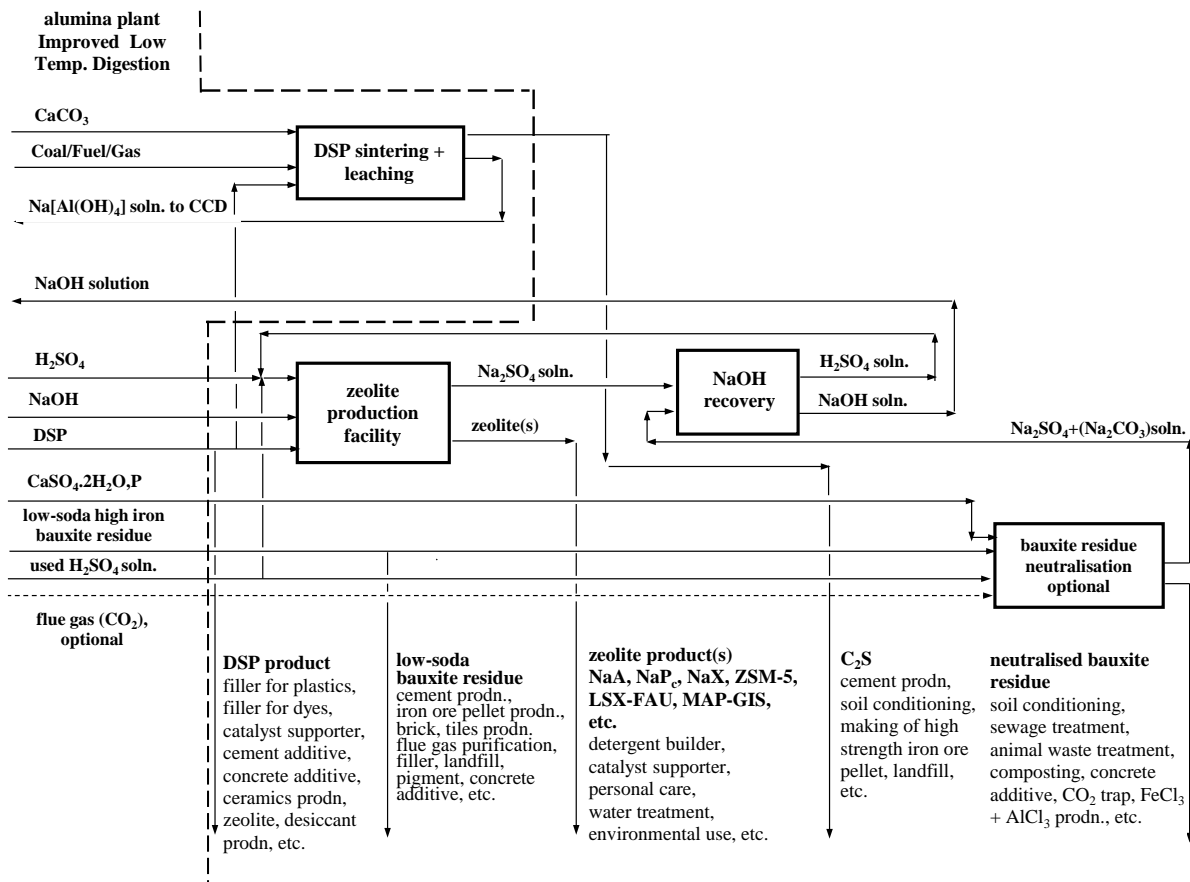


Figure 10. Downstream cooperation options and their connections with the ILTD Process [9].

## 12. Development Efforts to be Undertaken

- A Concept Study of the given alumina refinery and feedstock, possibly in alternatives (3 months)
- Laboratory (preferably bench scale) kinetic tests for the anticipated bauxite/laterite feedstock(s), possibly with the separation of bauxite residue (anticipated 3-6 months)
- Design, building, commissioning, and execution of the pilot scale implementation of the ILTD Process concept (anticipated 18-24 months)
- Techno-economic Feasibility Study for the ILTD Process concept (anticipated 6-9 months).

The laboratory (preferably bench scale) testing and the design of the pilot scale implementation of the ILTD Process can be carried out in parallel (3-6 months).

## 13. Benefits of the ILTD Process

The major benefits of the ILTD Process compared with the CLTD Process have been proven to be:

- 30-80 % savings in chemical NaOH losses depending on bauxite quality and process configuration
- some 3-7 % savings in bauxite consumption
- up to 20 % savings in process heat consumption
- 10-20 % higher digestion capacity
- 10-20 % higher liquor productivity during precipitation
- 10-30 % less low soda bauxite residue

- less silica and iron contaminants in the product (by 10-40 %)
- less scaling in the liquor preheaters (by 40-60 %), longer operating time and/or less heat consumption
- option of the recovery of the soda and the alumina content of DSP within the refinery and/or converting it to value-added products such as different zeolites [9]
- less degradation of the organic material in bauxite due to the minimum reactive OH<sup>-</sup> concentration and short digestion reaction time (estimated reduction of 50-70 %)
- enables viable processing of bauxite bodies with relatively high silica and/or relatively high boehmite and/or possibly with relatively high goethite content
- the utilization of low soda bauxite residue has a great potential of growth
- the composition of the low soda bauxite residue enables at least its partial utilization instead of its disposal
- the rehabilitation of a disposal site of a low soda bauxite residue is much faster and cheaper than that for the bauxite residue from a conventional process.

#### **14. Significance of the ILTD Process for Processing Low Grade Bauxites/Laterites**

It is deemed that latest calculations are convincing that the ILTD Process can convert significant part of the sub-economical resources to ores that can be processed in a viable way. Lyew-Ayee estimated the overall grand total of bauxite resources at 75-100 Gt [10]. He suggested a classification of bauxites for the Bayer process based on the mineralogical composition with constraints for the different classes (gibbsitic (< 5 % boehmite), mixed gibbsitic-boehmitic (5-20 % boehmite), boehmitic (> 20 % boehmite), diasporic (> 5 % diasporic). Bárdossy and Aleva estimated 40 Gt of bauxite reserves [11], out of that 14 Gt was classified to be sub-economic (probably due to their chemical/mineralogical composition) or hypothetical. The total amount of resources was estimated to be 54 Gt. Komlóssy estimates some 88-90 % of the reserves to be of gibbsitic type ores, of which, about 5 % are mixed gibbsitic-boehmitic bauxites [12]. Based on the sporadic information available, the amount of 10-25 Gt for the sub-economic resources, which could become feedstocks for alumina refineries where an updated ILTD Process is implemented is deemed to be a conservative estimate.

#### **15. Conclusions**

The Improved Low Temperature Digestion (ILTD) Process is considered to be a breakthrough in the development of the Bayer process, as far as its viability is concerned and also the economic use of resources that until now have been considered to be sub-economic.

The low soda bauxite residue is highly welcome in various industries, eg. the use in Portland cement clinker production and in the iron/steel industry, therefore, making such bauxite residue is a substantial step towards improving the sustainability of the Bayer process.

The alumina in aluminosilicates in bauxites (first of all in kaolinite) is correctly not considered to be available within the framework of the conventional Bayer process. However, when soda and alumina content are recovered from the DSP by-product of the ILTD Process by sintering, the alumina originally in kaolinite becomes extractable in a viable way. In this case, the higher R.SiO<sub>2</sub> content in bauxite causes minimal material and energy cost increase, which is the opposite to that found in the conventional Bayer process.

The DSP by-product could easily be processed further, recovery of its alumina and soda content by sintering is only one viable option. The C<sub>2</sub>S belite by-product of this process solution could be readily utilized in the cement industry, therefore the updated ILTD Process is deemed to be another significant step towards the sustainable production of alumina with no wastes to dispose of.

The ILTD Process concept calls for a shift of paradigm. The last sentence of the paper [4] is quoted here „Probably in the near future, silica in bauxites will not be considered as a constituent that is simply responsible for costs and many troubles but rather as one that can also be used to increase the economy of the Bayer process!”.

The ILTD Process concept is deemed to be ready for its commercialization.

## 16. Acknowledgements

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