ORGANIC REMOVAL AND ITS EFFECT ON PRECIPITATION AND ALUMINA

QUALITY

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ABSTRACT

The different processes developed for organic removal are evaluated based on a detailed review of the literature. The bauxite and technology related distribution of the organic compounds in process liquors of different alumina refineries are compared, completed by the balance of organics determined in an Hungarian refinery.

The following processes for organic removal are discussed in details: evaporation of the hydrate wash water, wet air oxidation, use of synthetic polymers.

Some laboratory tests related to the effect of the organics, especially humates and oxalate on agglomeration process are reviewed.

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1.0 INTRODUCTION

There is a general, common task for all alumina plants to control the inorganic and organic impurities enriched in the closed circuit of the Bayer process. /VERGHESE, 1987/ Proved methods are applied for the removal of inorganic contaminants /carbonate, sulphate, fluoride, phosphate, vanadate etc./ to avoid the reducing of liquor productivity and to minimize scalings and pluggings in the process equipment. For the control of soda /Na_2CO_3/ level the inside causticization during the red mud washing /BAKSA et al., 1986/ and the soda salt removal after crystallizing evaporation of the recycled liquor are available.

Organic materials or their degradation products /first of all humates and oxalate/ both reduce the liquor productivity and impair the quality /increasing Na_2O/ and physical properties of alumina /SANG, 1988; HOWARD, 1988/.

1.1 Methods for Organic Removal

Although a number of methods have been elaborated and published for organics removal and some of them, at least partially, are in operation, as well, no method has been found until now applicable effectively and economically under plant conditions everywhere. Aluminium Pechiney preferred barium-aluminate for carbonate and oxalate removal /US Patent 4,101.629/ Kaiser Aluminium recommended the proper addition of dawsonite /US Patent 4,036.931/, ALCOA Chemie /formerly Giulini/ and Kali und Salz A. G. used magnesium compounds, preferably kieserite for the precipitation of humates /Australian Patent 13267/76, US Patent 4,046.855, European Patent 188.268; - POLAND and TIELENS, 1983/, Showa Denko proposed the ignition of the liquor /US Patent 4,280.987/, Kiser and NALCO suggested the dosage of special cationic polymer for humates removal /US Patent 4,578.255/. All of the mentioned methods were found as ineconomic under the Hungarian conditions where the bauxite processed contains 1.0-1.5 % CO_2, however, 0.07-0.10 % organic carbon only. /MÁTYÁSI et al, 1986/

An other group of the methods recommended for organics removal applies the wet oxidation /YAMADA et al., 1981, THE et al., 1985/ at different temperatures, using oxygen or air /US Patent 4,581.208/, sometimes catalyst, e.g. Cu, as well /West German Patent 3,512.404/.Kaiser Aluminium /FOSTER and ROBERSON, 1988/ developed a partial wet oxidation process by in the liquor dissolved oxygen for destructuring of the high molecular weight organic compounds only /humates only/, however, the continuous operation could not been justified economically in spite of the successfull plant tests /US Patent 4,578.255/

Recently STUART /1988/ reported about the latest re-results achieved in Australia in the field of organics removal from Bayer spent liquors using manganese dioxide ore. The process needs further developing works.

It should be mentioned the methods applied for oxalate control especially /GNYRA and LEVER, 1979/. The behaviour of sodium-oxalate in the Bayer process is quite well known /BROWN, 1980/ and the methods for its removal are continuously under development /CHARTOUMI, 1988/.

Two methods for organics removal has been developed in Hungary, as well, namely the evaporation of the hydrate wash water rich in oxalate /US Patent 4,335.082/ and the wet air oxidation combined with sodium-carbonate and sodium-oxalate removal formed during the high temperature oxidation by means of the filtration of the salt obtained./British Patent 2,037.722/

1.2 The Aim of the Liquor Purification

CHIN /1988/ emphasized that the thermal decomposition of the organic impurities is the only broad-spectrum method for destruction of all organic impurities in a sidestream of spent liquor. Other liquor purification methods, such as adsorption on low-cost adsorbents /e.g. carbon/, evaporative crastallization, polymer addition or wet oxidation are selective for some harmful components of organics, only.

In spite of the technical and economic difficulties of liquor purification, the R&D activivity is focused on this question in the next future, as well. It is a common tendency to approach the "American" and "European" Bayer-variants each to other with the aim to produce sandy alumina at an increased /high/ liquor productivity. It means the increasing of the caustic soda concentration at the same sandy alumina quality on the one hand and the conversion of the floury alumina production into sandy or sandy-like one maintaining the high liquor productivity on the other hand /WINKHAUS, 1987/.

We are producing actually the traditional floury alumina in Hungary, therefore we have to deal with the liquor purification from point of view of the changing of product quality at a liquor productivity as high as possible.

2.0 ORGANICS IN PROCESS LIQUORS

The extraction and degradation of organics are depending on the bauxite processed and the digestion technology applied. Consequently, the problems arising by the organic content of the process liquors are also different and specific for each alumina refinery. The development of a sophisticated analytical system to measure and follow the changing of the

different organic species in the course of the Bayer process
including the control of the different methods for liquor
purification, was inevitable, therefore numerous work was
carried out for this purpose /e.g. LECTARD and NICOLAS,
1983; BRINDEL and LECTARD, 1983; GUTHRIE et al., 1984/.
Recently GROCOTT /1988/ reported about a fast analytical
method to determine the extractable organic carbon, oxalate
and carbonate from bauxite during digestion.

2.1 Distribution of Organic Compounds in Different Alumina Refineries

We have developed a method based on the gas-chromato-
graphic determination of the esterised organic com-
pounds. By means of this procedure the distribution of
organic compounds of different process liquors from
alumina refineries has been determined. The results
calculated on the same caustic soda concentration/256
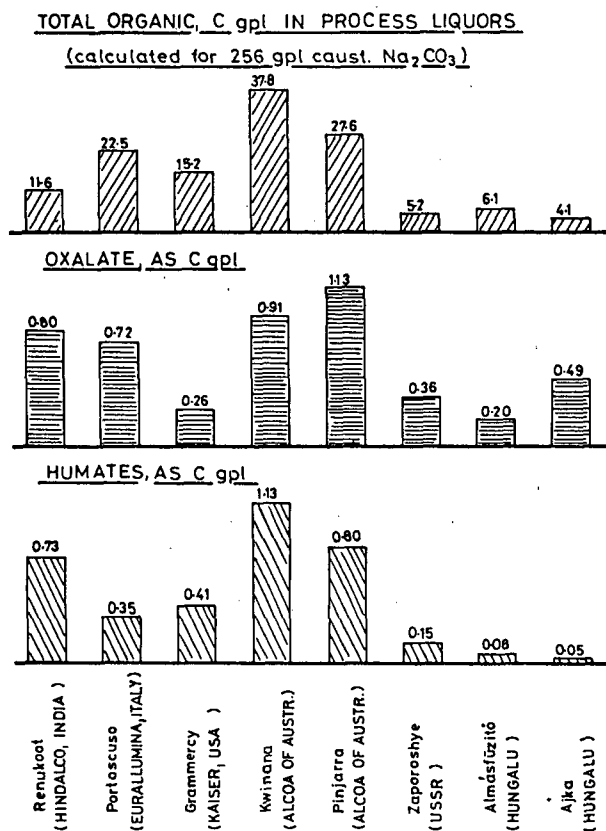gpl caustic soda as Na_2CO_3/ for better comparison are
plotted in Figure 1.

TOTAL ORGANIC, C gpl IN PROCESS LIQUORS
(calculated for 256 gpl caust. Na_2CO_3)

OXALATE, AS C gpl

HUMATES, AS C gpl

Renukoot (HINDALCO, INDIA)
Portoscuso (EURALLUMINA, ITALY)
Grammercy (KAISER, USA)
Kwinana (ALCOA OF AUSTR.)
Pinjarra (ALCOA OF AUSTR.)
Zaporoshye (USSR)
Almásfüzitö (HUNGALU)
Ajka (HUNGALU)

Figure 1. The total organic car-
bon content in the process li-
quors of different alumina re-
fineries.

The extremely high
total organic con-
tents, as well as oxa-
late and humate con-
tents of the Western
Australian process
liquors are characte-
ristic and can be ex-
plained by the very
high bauxite consump-
tion. The organic le-
vel of the refineries
processing European
karst bauxite is re-
latively low, however,
the oxalate content
can be increased here
also above the satu-
ration disturbing the
precipitation and
hydrate quality.

The distribution of
the different identi-
fied organic compo-
nents expressed as C,
in the same investi-
gated liquors rela-
ted to the total or-
ganic content /100 %/
can be seen in Fi-
gure 2. It is remark-
able that the "not
identified" part of the total organics is the highest
in the process liquors of refineries processing late-
ritic bauxite at low temperature digestion /Kwinana
and Pinjarra plants/.

We are supposing that this group of organics is less reactive, much more stable than others which can be confirmed by the fact that after wet air oxidation the identified part of the total organic content is increasing, due to the destructuring. Nevertheless, this large amount of the not identified organics can cause quite significant uncertainity in our evaluation related to both the effect and control of organics. The simulation of this "black box" organics is also impossible A lot of efforts should be made to clear this problem.
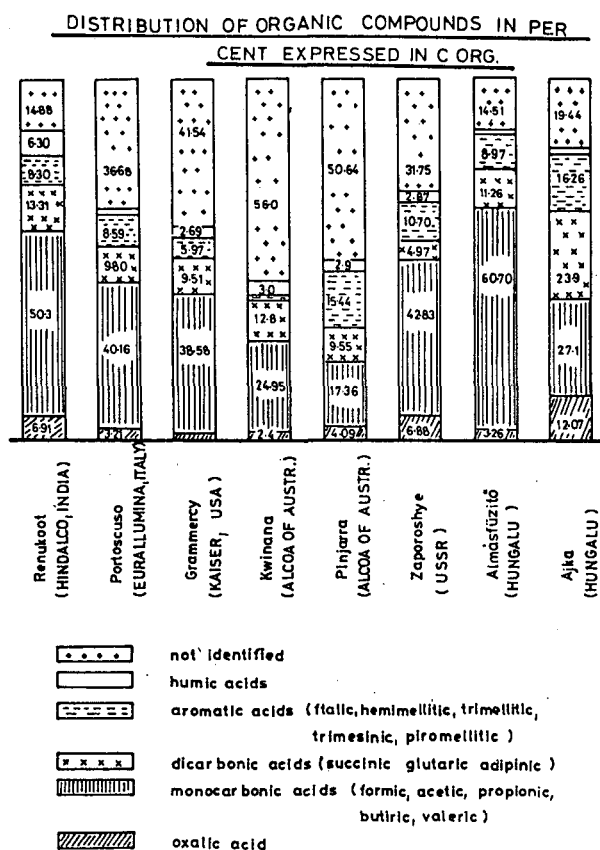

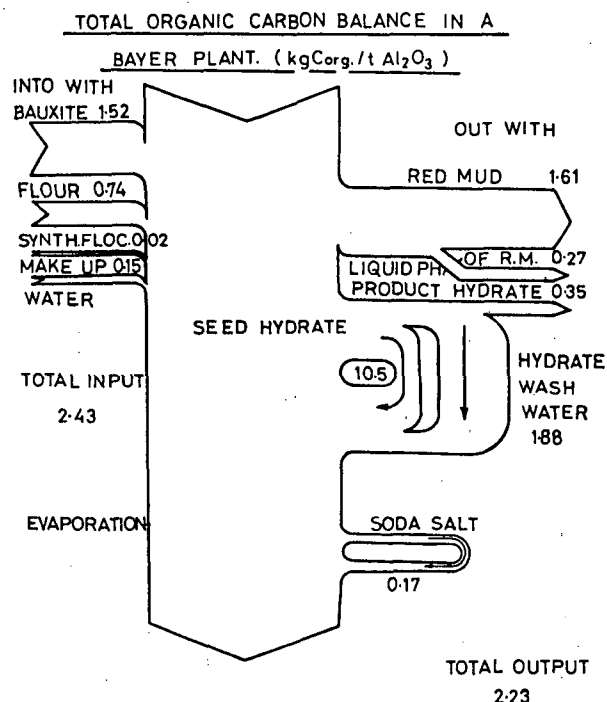Figure 2. Distribution of organic compounds measured by gas-chromatography.

2.2 Organic Balance of the Hungarian Almásfüzitő Refinery

The balance of the total organic content in the Almásfüzitő Refinery /Hungary/ is demonstrated in Figure 3. and in Table 1.

Table 1. Balance of the total organics in Almásfüzitő Refinery

Input with	C_{org} — $\frac{kg/t \ Al_2O_3}{}$	Organic content in
Bauxite	1.52	
Flour	0.74	
Synthetic flocc.	0.02	Soda salt 0.17
Make up water	0.15	Hydrate
Total input	2.43	wash water 1.90
Output with		Seed hyd-
Red mud	1.61	rate 10.50
Liquid phase of red mud	0.27	
Product hydrate	0.15	
Total output	2.22	
Remaining in cycle	0.21	

The most important source of organics is the bauxite and the largest amount of organics leaves the cycle by red mud and product hydrate. In the investigated period about 10 % /0.21 k per tonne of alumina/ remained in the cycle increasing the level of organics. The enrichment of the organic content can be observed in seed hydrate /10.5 kg/t alumina which is 10 per cent of the total organics present in the whole Bayer cycle/ and the hydrate wash water /1.90 kg/t alumina/ The organic content of the seed hydrate decreases the efficiency of the precipitation by blocking the active places on the surface of the hydrate crystals.

Figure 3. The balance of the total organics in the Almásfüzitő Refinery in 1979.

In Table 2. the qualitative and quantitative distribution of the identified organic compounds of the strong liquor of Almásfüzitő Refinery is summarized, based on the gas-chromatographic determination.

Table 2. Organic compounds identified in the strong liquor of Almásfüzitő Refinery /in C_{org}/

Na_2O caustic				150 gpl
as Na_2CO_3				256 gpl
C_{org} total				6.12 gpl
Formic acid	0.39 gpl C	Adipic acid		0.03
Acetic acid	2.81	Pimelic acid		0.02
Propionic acid	0.27	Phtalic acid		0.06
Butylic acid	0.08	Hemimellitic acid		0.07
Valeric acid	0.16	Trimellitic acid		0.19
Oxalic acid	0.19	Trimeric acid		0.14
Benzoic acid	0.01	Piromellitic acid		0.09
Succinic acid	0.48	Humic acids		0.08
Glutaric acid	0.15	Total identified		5.22
		Not identified		0.90

In Table 3. the distribution of the identified organic compounds of the seed and product hydrates are compared /expressed in C_{org}/ It can be seen that the organic compounds can be washed out very effectively. The seed hydrate is not being washed actually in our practice because this operation is not economic due to the high

seeding ratio /more than 3.0/, although it would be ad-
vantageous to produce coarser alumina.

Table 3. Organic compounds identified in the seed and
product hydrates

Organic compound	in C_{org} kg/t alumina	
	in seed hydrate	in product h.
Humic acids	0.13	0.02
Oxalic acid	2.10	0.08
Formic acid	0.19	0.01
Acetic acid	2.30	0.10
Aliphatic acids	4.20	0.13
Aromatic acids	0.90	-
Total identified	9.82	0.34
Not identified	0.68	-
Total	10.50	0.34

3.0 METHODS FOR ORGANICS REMOVAL INVESTIGATED IN HUNGARY

One of the possibilities for reducing organic level is the
use of synthetic flocculants instead of natural ones /flour,
starch/. In our earlier practice about 30-35 % of the total
organics was originated from flour. We are applying actu-
ally in one of our alumina plants /MOTIM Works/ ALCLAR 600
flocculant only without any flour /PEARSE and SARTOWSKI,
1984/. The organic level decreased by 1.3 gpl in the strong
liquor after one year such operation already. In other two
plants we are using now ALCLAR polymer and flour together
for settling, however, we are running there without control
filtration.

The soda salt removed after crystallization-evaporation con-
tains, depending on the organic content of the liquor, 0.5
to 1.5 per cent $C_{organic}$ which can be destroyed by ignition
at 500-600 °C before causticization. This process is also
costly, therefore could be applied in critical situation,
only. Nowadays we are dealing with three other methods, too.

3.1. Evaporation of Hydrate Wash Water

By concentrating the hydrate wash water containing a-
bout 2.0 gpl C_{org}. and 30-40 gpl Na_2O caustic to a
concentration 5 to 7 times as high as the original one
/up to about 200 to 220 gpl caustic Na_2O/ 0.4 to 0.8
kg organic carbon can be removed per tonne of alumina.
The salt precipitated during the evaporation of alca-
line hydrate wash water contains 13-15 % organic car-
bon and 70-80 % of this is present as sodium oxalate.
The technological flow-sheet of the process applied
at Almásfüzitő Refinery can be seen in Figure 4. The
process is very effective for oxalate removal, however,
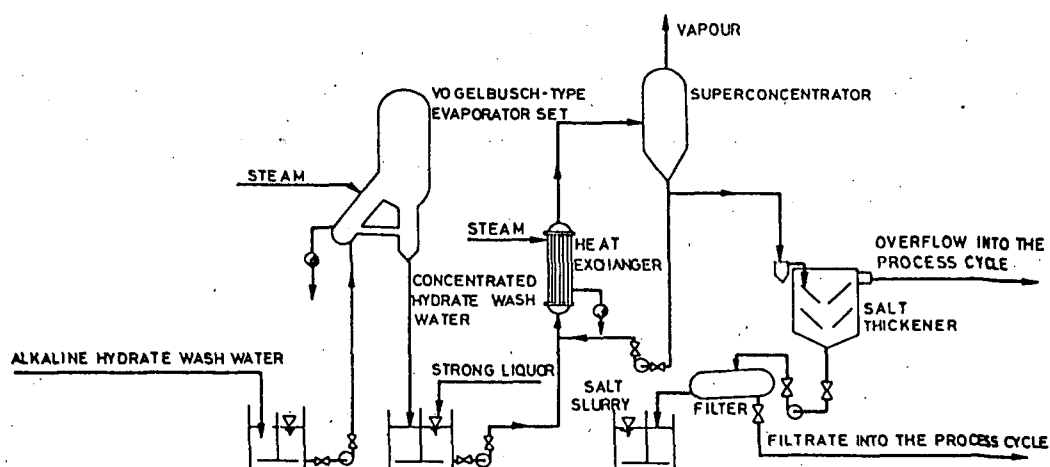5-10 % of other organic compounds can be removed, only.

Fig. 4.

TECHNOLOGICAL FLOW-SHEET OF ORGANICS REMOVAL
FROM HYDRATE WASH WATER

3.2 Wet Air Oxidation

The wet air oxidation does not remove the organic com-
pounds from the process liquor but partly destroys them
within the liquors /MÁTYÁSI et al., 1986/ The organic
compounds can be oxidized partly to sodium carbonate,
partly to low molecular weight organic sodium salts,
which are /except for sodium oxalate/ usually less harm-
ful than the higher molecular weight compounds.

Common batch laboratory tests were carried out in our
institute /ALUTERV-FKI/ in Budapest and in FNE, Frei-
berg, GDR /Forschungsinstitut für Nicht-Eisenmetalle/
in order to test this process and to develop it for
commercial use. The laboratory tests were carried out
in agitated laboratory autoclaves, firstly batchwise,
thenafter in continuous operation and using different
reactor types /autoclave, spray reactor, bubble column/
as well. The process is very effective to destroy /de-
structure/ the high molecular weight humates. This is
demonstrated in Figure 5.

In the next phase of development a 100 l batch auto-
clave has set up at Almásfüzitő Refinery. The results
confirmed that the yield of organics removal may be in-
creased significantly by means of separation of thede-
structured organic salts /mostly oxalate/, together
with the precipitated sodium carbonate salt.

In order to the upscaling an 1 m^3/h capacity, continu-
ous pilot-scale system has been constructed in Almás-
füzitő by the Alumina Plant and ALUTERV-FKI. The flow-
sheet of the system is shown in Figure 6. The pilot
plant consists of the following characteristic equip-
ment: Membran piston pump /Hauke/ with a capacity up to

1000 l/h and max. pressure of 75 bar; an 0.6 m^3 auto-
clave with a special agitator serving for the thorough
mixing of the liquor with compressed air delivered by
a two-step air compressor /max. capacity 70 Nm^3/h, max.
pressure 75 bar/. FNE experts were also participated

in the setting up of the
system and in its operation.
As a latest development the
pilot plant has been comp-
leted by bubble columns in-
stead of special stirrer as-
sisted autoclave. The later
system is more convenient
for operation. The results
obtained in the pilot plant
justified the laboratory re-
sults and confirmed the app-
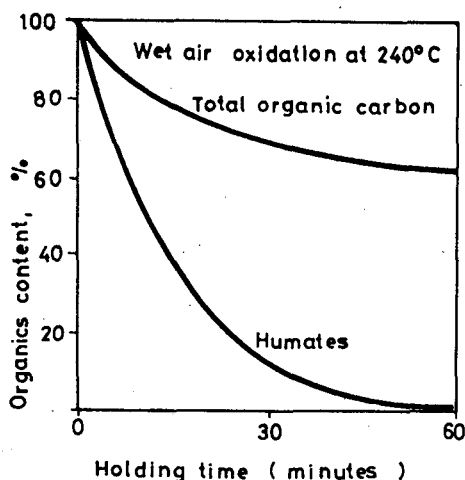licability of the method
for plant operation.

Figure 5. Lab. scale wet
air oxidation at 240°C in
the process liquor of Al-
másfüzitő refinery. Caust.
soda conc. 308 gpl as so-
dium-carbonate, A/C=0.29,
initial total Corg. 9.6
gpl, initial humic acid
content 0.33 Corg gpl.

Based on the laboratory
tests carried out with the
process liquor of Renukoot
refinery /HINDALCO/ has been
recommended to set up a
plant scale facility for wet
air oxidation of 40 m^3/h
strong liquor designed by
ALUTERV-FKI. The wet air o-
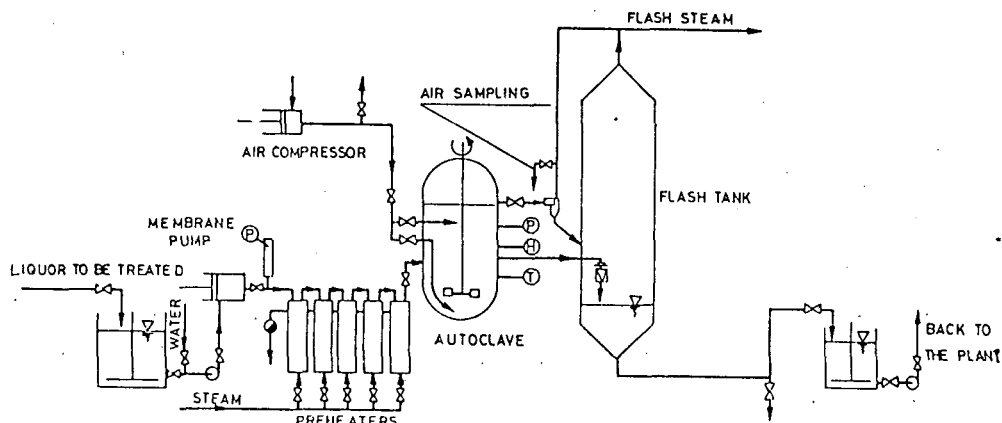xidation plant unit at Renu-
koot has been put into ope-

Fig. 6.

TECHNOLOGICAL FLOW-SHEET OF THE PILOT SCALE
WET AIR OXIDATION SYSTEM

ration at the end of 1986 by common efforts of Indian
and Hungarian experts and has been developed and modi-
fied sometimes until now in a good cooperation between
plant-experts and designers. During the first year of
operation /in 1987/ the total organic carbon content of
the circuit decreased by about 20-25 per cent and the

humate content by about 50 per cent. In spite of these results big difficulties have been arised by the insufficient filterability of the oxalate and fluoride rich fine-dispersed salt formed after flashing and cooling of the oxidized liquor. These unexpected difficulties at filtration have not been observed during the laboratory process simulation. Consequently, the $Na_2C_2O_4$ content present in the liquor after the oxidation /4-5 gpl sodium-oxalate at 280-320 gpl caustic soda concentration as sodium-carbonate/ could be reduced by filtration partially only, because 1/3 part of the full stream only could be filtered by means of the available designed equipment /Fundabec filter/ in order to decrease the $Na_2C_2O_4$ content down to 2.0-2.5 gpl at 70 $^{\circ}$C. As a result, the oxalate level in the Bayer cycle has been slightly increased hindering the further increasing of the caustic soda concentration and liquor productivity. Actions are going on to improve the separation of the oxalate rich salt /e.g. by means of centrifuging/. We are convainced that the problems will be avoided by means of the common efforts will be taken on behalf of our Indian colleagues and ourself.

It should be mentioned that at Renukoot refinery is not existing any process for oxalate control which is required applying any wet oxidation method with partial destructuring of organic compounds. According to our concept the oxalate control should be fulfilled by the separation of the oxalate-fluoride-carbonate containing salt formed, therefore we have to pay much more attention to the removal of the oxalate salt after oxidation and cooling, in the future and on site pilot plant tests are recommended, as well. In spite of some difficulties, the wet oxidation process is a very effective means for humate destruction.

3.3 Organics Removal by Synthetic Polymers

As a latest development in the field of organics removal should be mentioned the addition of synthetic polymers /cationic/ to form insoluble compounds with humates. CYANAMID /European Patent 211.338/, NALCO Chemical with Kaiser /US Patent 4,578.255/ and ALLIED COLLOIDS have developed special chemicals of similar behaviour which should be added in an amount of 30-50 g/m^3 diluted slurry.

We have carried out informative plant tests at Almásfüzitő Refinery with Magnafloc 369 /ALLIED COLLOIDS chemical/ when at the addition of 10 g/m^3 8-9 %, at the addition of 50 g/m^3 from this reagent up to 25 % decreasing of coloring materials have been observed. As an optimum dosage could be preferred 20-30 g /m^3 diluted slurry.

All these reagents are selective for humates and have a limitation related to the humate content can be decreased, however, there are advantageous from point of view of the minimum investment costs.

It is also remarkable that NALCO Chemical developed an other new chemical, as well, to produce coarser alumina and to remove sodium oxalate by means of an acrylic acid polymer /US Patent 4,608.237/.

The synthetic polymers have a good potential both for organic removal and for the conversion of floury alumina production to sandy one at high liquor productivity. It is to be emphasized that in contrary with oxidation, the polymers can remove organics in their original form, do not cause any decausticization and destructuring. As a main direction for further activity in this field can be preferred to develop chemicals for removal of total organic content, at least suitable ones to decrease significantly the amount of both humates and oxalate.

4.0 THE EFFECTS OF ORGANIC IMPURITIES ON PRECIPITATION

The simulation of the effect of organic impurities on precipitation is combined with big difficulties due to the interaction among the different organic species influencing non-additively the solubility of the different organic and inorganic constituents of the given liquor and their adsorption on the hydrate surface during the precipitation.

The only method can be preferred is to use plant liquors containing the full spectrum of contaminants and to add to this basic solution the different organic components to be studied.

The aim of our laboratory tests was to investigate the effect of the organic impurities, first of all that of the oxalate and humates, on the agglomeration stage in order to prepare the installation of this operation at Almásfüzitő Refinery to produce there coarser alumina.
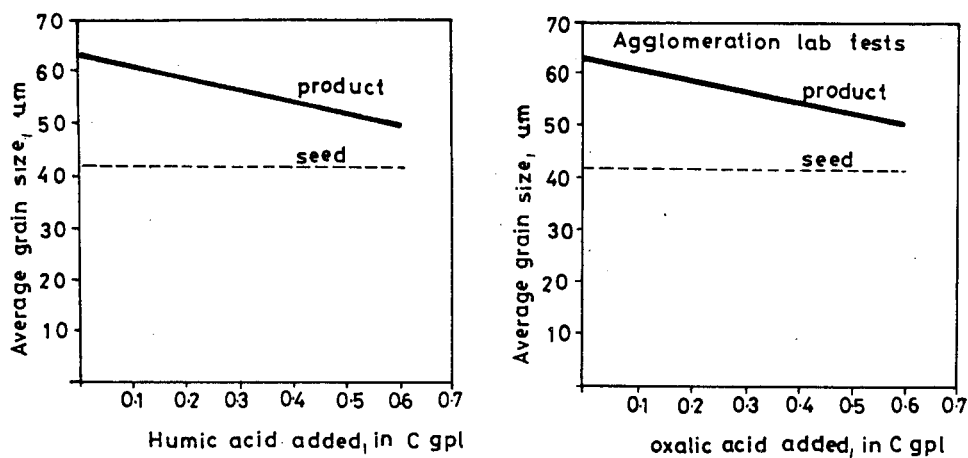

Figure 7. Agglomeration laboratory tests with the process liquor of Almásfüzitő Refinery, at 70 °C, with 0.3 seeding ratio and 8 hours retention time. Initial liquor: caustic soda: 222 gpl as Na_2CO_3, A/C=0.64.

In Figure 7. are plotted the effects of humate and oxalate

addition on the agglomeration, respectively. The attrition
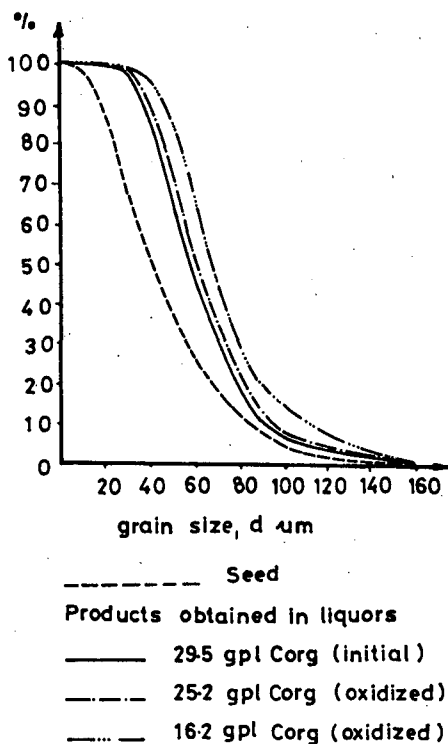of the products obtained has been increased in both cases,
as well.

Figure 8. Laboratory agg-
lomeration tests with the
process liquor of Kwinana
Refinery, at 72 °C, 10 h,
0.3 seeding ratio. Ini-
tial liquor: 193 gpl ca-
ustic soda as Na$_2$CO$_3$,
A/C=0.65.

The effect of the wet air oxida-
tion on the agglomeration has
been studied, too. The process
liquor of Kwinana Refinery /Aus-
tralia/ has been selected for de-
monstration. The results are
plotted in Figure 8. Although
the seed hydrate has not been
washed, the results show clearly
the advantages of the decreased
organic level achieved by labo-
ratory wet air oxidation.

Our laboratory tests confirmed
that each gpl decreasing in the
total organic content can in-
crease the liquor productivity
by 1-2 kg/m^3 Al$_2$O$_3$ precipitated,
using diluted or oxidized liquors
of similar organic spectrum.

Our laboratory investigations
and plant experiences confirmed
the published data related to
the close correlations between
the organic content precipitated
on the hydrate surface and the
grain size distribution, as well
as the Na$_2$O content of the alu-
mina produced. /SANG 1988,
HOWARD 1988/.

5.0 CONCLUSIONS

The problems arising by the organic content of the process
liquors are different and specific for each alumina refi-
nery. The use of a sophisticated analytical system to cont-
rol the different organic species in the Bayer cycle is
inevitable. Considering the methods developed and partly
used for organic removal in different refineries can be
stated that no method has been found until now which could
be applied effectively and economically under plant condi-
tions everywhere, because they are selective for given or-
ganic compounds only, furthermore bauxite and technology
related.

The evaporation of hydrate wash water, the wet air oxida-
tion and the application of inorganic and organic reagents
for organic removal were studied in Hungary. The wet air
oxidation is very effective for the destructuring of hu-
mates, however, it should be combined with removal of the
organic salts /mostly oxalate/ formed during oxidation.
Special synthetic polymers have a good potential for orga-

nics removal, in the future.

The simulation of the precipitation process should be carried out with plant liquors considering the non-additive influence of the organic compounds. It has been confirmed that the most harmful organic components are humates and oxalate.

6.0 ACKNOWLEDGEMENTS

The authors wish to express their appreciation to all co-workers, especially to Prof. S. Ziegenbalg and dr. D. Löwe /FNE, Freiberg/, dr. J. Mátyási, Mr. L. Révész and Mr. F. Tóth /Almásfüzitő Refinery/, dr P. Siklósi and Miss Zs. Kazár /ALUTERV-FKI/ taking part in the development and testing work for their efforts. Special thanks to our Indian colleagues at Renukoot Refinery /HINDALCO/ for their work and good cooperation.

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