

## Micronised Ilmenite - A New, Intermediate Weight Material for Drilling Fluids

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### Abstract

The primary function of a weight material is to increase the density of a drilling fluid. Since the 1920's, barite has successfully fulfilled this function due to its availability, cheapness, high density & relative inertness. However, there have been a number of changes recently, which suggest a need for alternative weight materials:

- 1) The global availability & supply of high grade ( $\geq 4.20$  sg) barite is diminishing rapidly
- 2) There is a need for weight materials, which provide other properties such as to facilitate ECD management, sag control, formation damage mitigation etc

There are a few weight materials such as manganese tetraoxide<sup>1,2</sup> or treated micronised barite<sup>3</sup> which satisfy some or all of these demands. They provide excellent, extra properties compared to barite, but such a high performance is not always needed for a particular application, the volumes are limited & they are more expensive. There is a need for alternative weight materials, which can provide better performance & cost, intermediate between barite & the high end weight materials which require extra logistics and handling needs.

This paper describes a new micronised, ilmenite, which offers significant advantages in the control of ECD, sag & formation damage. Tests show how it is used to create fluids with stable rheology with low PV's & gel strengths, even under HPHT conditions, for both water & non-aqueous based fluids.

It is denser than barite, has a better environmental profile, is highly acid soluble & is easy to recycle.

Accordingly, it provides a viable alternative product, intermediate between barite & the more expensive & specialised weight materials.

### Introduction

There is a reduced availability & increasing price of  $\geq 4.20$  sg barite, which is approaching a critical point. 6 years ago the

recognition of diminishing supplies led the API to investigate a second standard for barite, 4.1 sg. This grade, which was introduced over 2 years ago, was intended to enable the continued supply of barite (especially in the US) by approximately 11 years with minimal expenditure. 4.1 sg barite could be used for fluids at  $< 14$  ppg<sup>4,5</sup>. This takes into consideration the increased impurities, which may mitigate against using lower grades of barite for heavier fluid applications. However, a number of factors have altered this situation:

- 1) China produces over 50% of the world's barite. Chinese exports have reduced by approx 1 million tonnes in the past year; almost 15% of world supply
- 2) India initiated a price rise of *ca* 75% in 2011. China has followed this, increasing prices incrementally
- 3) The Yuan has appreciated against the US dollar by around 25% in the past 7 years
- 4) Logistics costs have risen with increased oil prices

This has resulted in barite prices more than doubling in the past year, tripling in the past 5 years & still continuing to rise. Therefore, the barite market sees both diminishing supplies of high grade material & increasing prices. The result of this is a need to rethink the approach of using  $\geq 4.20$  or 4.10 sg barite as the only or even primary weight material in drilling fluids.

The second consideration is that of the requirements of modern drilling operations, which impose greater demands on the functions & properties of drilling fluids & the influence the weight material exerts on these:

- More difficult drilling environments & operations such as HPHT, ERD, deepwater etc., require tighter ECD management & sag control. Good, stable control of rheology is needed, especially at high temperatures & pressures. API Barite is often incapable of providing the desired performance
- Fluids need to be non-damaging to prevent productivity impairment in the reservoir

- Increasingly stringent HSE regulations require “green” chemicals & more recycling of fluids to improve sustainability

Clearly, there is a need to introduce alternative materials, which can supplement the use of barite by e.g. increasing the density when 4.1 sg barite is used or by providing the increasing performance required of modern drilling fluids.

Over the past 90 years many materials have been suggested & tested as potential weight materials for drilling fluids e.g. hematite, itabirite, ilmenite, siderite, celestite<sup>6,7,8,9,10,11</sup> etc., but all have had attendant issues such as abrasivity, insufficient density, cost, magnetic influence etc. It is interesting to note that the use of hematite preceded that of barite. The use of barite became pre-eminent largely due to its lower cost & ready availability within the US. It seems unlikely that a material, can be found to wholly replace barite. What other materials could provide the density, volumes, cheapness etc? Therefore, we need to reconsider how we use the barite reserves we have left, how to differentiate between other available weight materials & how we apply their use e.g. it is conceivable to consider the use of several weighting agents on the same drilling project.

## Ilmenite

The potential use of ilmenite in drilling fluids has been known for many years, but the first sustained attempt at using it to densify drilling fluids was in 1979 in Norway<sup>12</sup>. The failure to continue its use at that time occurred mainly because, like hematite, it was abrasive & resulted in an unacceptable damage to both surface & downhole equipment. From 1993,<sup>13,14,15,16</sup> further tests were conducted on ilmenite leading to the understanding that if the coarse particles >45µ were maintained below 0.5% w/w then ilmenite exhibited no worse abrasion than that of API barite. However, whilst the use of this ilmenite with a D<sub>50</sub> of 10-15 µ continued in the North Sea (mainly in Norway) it never really took off. The reasons for this are quite simple. Ilmenite was used as a substitute for API barite & with similar performance, but over the years the price increased to where ilmenite was over twice the cost of barite. The advantages of ilmenite became less obvious as the price differential increased.

Over the past 10-12 years there has been increased interest in speciality weight materials. This has resulted from the need to improve the performance of drilling fluids in a number of ways:

- 1) Provide low rheology (low plastic viscosity) particularly in high density fluids >16 ppg to provide good ECD management

- 2) Show low settling tendency providing low sag
- 3) To be easily removable from the reservoir by mechanical or chemical means so as not to impair productivity
- 4) To be cost effective
- 5) To have an acceptable HSE profile

Two main materials have come into use to address these requirements. Manganese Tetraoxide<sup>1,2</sup> & treated micronised barite<sup>3</sup>. Both materials have been shown to provide many of the performance improvements needed by the industry. However, they are in limited supply & significantly more expensive than barite. Therefore, whilst they fulfill the objective of improving fluid performance they cannot replace the use of barite but can supplement it, only to a small degree.

## Micronised Ilmenite

The use of ilmenite as a direct substitute for API barite failed, not because the ilmenite offered poorer performance, but because it did not offer better value in that particular form. A new & optimised ilmenite offers significantly better performance & value.. This is a dry ilmenite, which has been micronised to a particle size distribution (PSD) with a D<sub>50</sub> of approx 5µ & a D<sub>90</sub> of <15 µ.

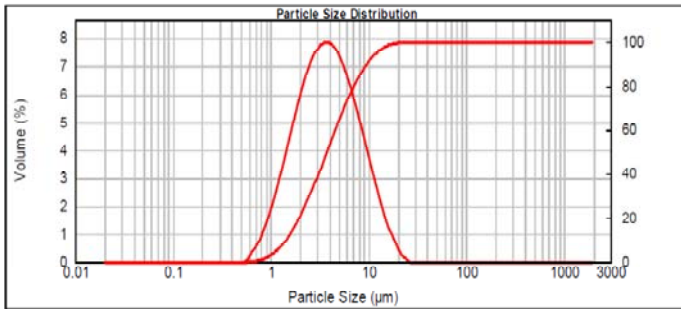
The morphology of the micronized ilmenite was measured by scanning electron microscope as shown in Figure 2 and by image analysis using the Morphologi®G3 particle characterization system from Malvern® Instruments. The imaging analysis shows that the particles are almost spherical with a total circularity of 0.9. The circularity is a parameter that describes the particle morphology or shape and is determined by the following equation:

$$\text{Circularity} = 4\pi A / P^2$$

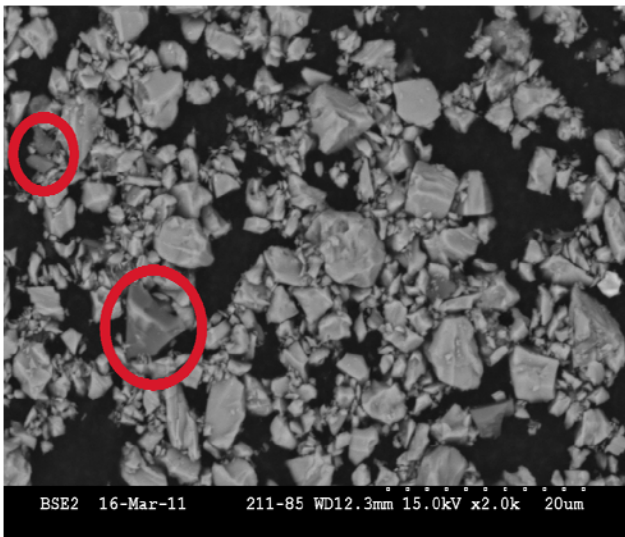
where A is the particle area and P is its perimeter. Such high circularity is of great interest from a rheological point of view as it provides fluids with low plastic viscosity.

PSD in µm			BET	Density	pH	Magnetite content
D10	D50	D90	in m <sup>2</sup> /g	in kg/l		Wt.-%
1.7	5	12	1.6	4.56	8.5	<0.3%

**Table 1: Typical Physical Properties of Micronised Ilmenite**



**Figure 1: PSD Analysis of micronised ilmenite**



**Figure 2: Photomicrograph of Micronised Ilmenite**

When ilmenite was first used in the early 1980's a number of issues were noted:

- 1) Higher abrasion of surface equipment such as mud pumps or transfer lines & downhole tools
- 2) Magnetic interference, which could affect MWD tools or electric logging tools such as NMR

Work done on the re-introduction of ilmenite in the 1990's determined how to resolve these problems:

- 1) To reduce abrasion to the same or lower level as API barite the coarse particles had to be removed such that particles of  $>45 \mu$  were kept below 0.5% w/w
- 2) To restrict magnetic dampening/shielding effects to the same or lower levels than barite the concentration of magnetite in the processed ilmenite needed to be kept  $< 0.3\%$  w/w

With a density of *ca* 4.6 sg ilmenite is approximately 10% heavier than barite, so it offers proportionally fewer solids in

the mud. This leads to more stable rheology & can also improve the rate of penetration when drilling.

Ilmenite is very hard, measured at 5-5.5 Mohs. Removing the problem of abrasiveness by limiting the amount of coarse particles means that the hardness of ilmenite becomes an advantage as fluids containing ilmenite are then easier to recycle because there is little change in the PSD of the ilmenite.

Ilmenite has an excellent HSE profile. It is rated PLONOR (Poses Little Or No Risk) in the North Sea. It is not mobile, does not persist or bioaccumulate & has no ecotoxicity. It has no regulatory risk phrases. Studies have further showed that the heavy metal contamination of Ilmenite is less soluble and bio-available than for Barite. "The study showed that flatfish fed with fish feed spiked with ilmenite, displayed no acute effects such as mortality or reduced feeding rate (growth). Fish exposed to barite showed increased concentrations of lead and barium in liver and blood. No such effects were observed for fish exposed to ilmenite."<sup>18</sup>

The analysis of using ilmenite in Norway showed that it resulted in:

- Reduced sag potential
- Improved hole-cleaning
- Longer lifetime of the drilling fluid
- Land-based cuttings handling
  - Burning with barite problematic
  - Burning with ilmenite straightforward
  - Less heavy metals from ilmenite if deposited
- Better shaker screen performance
  - Improved sand tolerance

Reducing the mean PSD of ilmenite to a  $D_{50}$  of *ca* 5  $\mu$  results in other significant improvements:

- Lower viscosities, so reduced pressure losses & pumping requirements can be achieved
- Reduced sag (settling capacity)
- Higher densities (fewer solids needed)
- Increased recyclability as the hardness means particle size is maintained
- Increased acid solubility for wellbore clean-up

### The Properties of Fluids Containing 5 $\mu$ Ilmenite

Previous work has shown<sup>1,2,3</sup> that micronising weight materials can lead to beneficial properties such as reduction in PV's & reduced sag. What are the effects of micronising ilmenite?

A series of tests comparing the results of 1 $\mu$  manganese tetraoxide vs 5 $\mu$  ilmenite vs API barite at *ca* 15  $\mu$  are shown in tables 2 to 7.

As might be expected we see a gradation of physical properties represented by the different PSD's of each weight material:

After static heat aging a 13.3 ppg OBM gave PV's of 18, 20 & 27 lbs/100 sq. ft for  $Mn_3O_4$ , ilmenite and barite respectively.

After static heat aging a 17.2 ppg OBM gave PV's of 34, 56 & 58 lbs/100 sq. ft for  $Mn_3O_4$ , ilmenite and barite respectively

After static heat aging a 19.2 ppg WBM gave PV's of 20, 44 & 54 lbs/100 sq. ft for  $Mn_3O_4$ , ilmenite and barite respectively

This reduction in PV represents a reduction in ECD of the fluid when used in the field.

A commensurate reduction in static sag can also be seen:

After static heat aging a 13.3 ppg OBM gave static sag of 0.511, 0.506 & 0.540 for  $Mn_3O_4$ , ilmenite and barite respectively.

After static heat aging a 17.2 ppg OBM gave static sag of 0.506, 0.508 & 0.522 for  $Mn_3O_4$ , ilmenite and barite respectively

After static heat aging a 19.2 ppg WBM gave static sag of 0.501, 0.511 & 0.570 for  $Mn_3O_4$ , ilmenite and barite respectively

The fluid loss of the micronised weight materials is generally higher than that of API barite. This is because of the much wider PSD seen in API barite. The PSD's (from  $D_{10}$  to  $D_{90}$ ) of both  $Mn_3O_4$  & the 5  $\mu$  ilmenite are very narrow. This leads to less tight particle packing & needs to be adjusted for when formulating lab muds. In actual drilling use, as seen with field studies of Manganese Tetraoxide, the addition of other LGS from drilled cuttings tends to widen the PSD of the solids in the fluid & leads to better particle packing, a less permeable filter cake &, consequently, a lower fluid loss. The fluid loss of a micronised ilmenite drilling fluid can be improved in several ways such as by blending with barite,  $Mn_3O_4$  or calcium carbonate. It can also be improved by optimizing the fluid loss package using different fluid loss additives.

### Acid Solubility of 5 $\mu$ Ilmenite

Ilmenite is readily soluble in most acids used for wellbore clean-up or stimulation.

However, it is notable that decreasing the PSD of ilmenite increases both the degree & rate of dissolution..

To demonstrate the effect of ilmenite particle size on the dissolution rate, three different grades of ilmenite, with an average size ( $D_{50}$ ) of 5, 18 and 70 $\mu$ m, were tested. The solubility of ilmenite was tested at 100°C in two media with different HCl concentrations of 10 and 20 % w/w of solution, respectively. In the 20 % w/w solution an additional 0.5mol methanol was added to enhance the solubility.

#### Solution I (10%HCl i.e. 2.83mol/l)

40.5g conc. HCl (37%) + 109.5g water + 7.5g ilmenite

#### Solution II (20%HCl i.e. 5.9mol/l)

81g conc. HCl (37%) + 66g water + 3g methanol+ 7.5g ilmenite

The Figures 3 & 4 below show the ilmenite solubility in the two solutions after 180 mins. The dissolution is described as Fe in mg/l. It is clear from the diagrams that the dissolution rate is proportional to the size of the particle. The dissolved Fe is in a logarithmic relation to the particle size or the surface area .

Importantly, the 5  $\mu$  ilmenite has a solubility of > 90% suggesting an excellent capacity for remediation . This distinguishes it from most other weight materials & particularly barite in any form.

### Conclusions

There is a growing need to find alternative weight materials to barite. This is because the availability of API grade barite is diminishing & needs to be managed to preserve sustainability in the long term. There is an increasing need to provide weight materials with better performance than barite, which will enable ECD management, sag control etc.

A micronised, 5 $\mu$  ilmenite has been produced , which, after testing ,shows significantly improved fluid properties when compared to API barite. The 5 $\mu$  ilmenite offers fluid performance intermediate between API barite & a high end weight material such as manganese tetraoxide. Therefore, the 5 $\mu$  ilmenite can be used either to supplement the use of barite or to replace it when increased performance is required in WBMs, NAFs .

## Nomenclature

*API = American Petroleum Institute*  
*CTD = Coil Tubing Drilling*  
*ECD = Equivalent Circulating Density*  
*ERD = Extended Reach Drilling*  
*HPHT = High Pressure High Temperature*  
*HSE = Health, Safety & Environment*  
*LGS = Low Gravity Solids*  
*MT = Metric Ton*  
*MWD = Measurement While Drilling*  
*NMR = Nuclear Magnetic Resonance*  
*OBM = Oil based mud*  
*PSD = Particle Size Distribution*  
*PV = Plastic Viscosity*  
*SG = Specific Gravity*  
*TTRD = Through Tubing Rotary Drilling*  
*WBM = Water Based mud*  
*YP = Yield Point*

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Chemicals	Mass in (gm)			Mixing time (min)
Mineral oil	301,0	301,0	301,0	
Amine based wetting agent	12	10	8	5
Fatty acid based emulsifier	9	9	9	5
Organophilic clay	5	5	5	10
Lime	9	9	9	5
Lignite based fluid loss agent	18	18	18	5
Polymeric fluid loss agent	2	2	2	5
CaCl2	25	25	25	15
Water	95,0	95,0	95,0	
Manganese Tetraoxide-(Mn3O4)	528			10
Ilmenite (FeTiO3)		556		10
Barite (BaSO4)			603	10

**Table 2: Recipe and mixing procedure of 13.3ppg OBMs with Mn<sub>3</sub>O<sub>4</sub>, ilmenite and barite**

Test results			Mn <sub>3</sub> O <sub>4</sub>		Ilmenite		API Barite	
			BHR	AHR	BHR	AHR	BHR	AHR
Temperature °C		°C	150	150	150	150	150	150
Period static aging		Hours		16		16		16
<b>Rheology: Temp °C</b>			50	50	50	50	50	50
	600 rpm		55	45	51	43	60	61
	300 rpm		34	27	32	23	37	34
	200 rpm		27	21	25	17	28	23
	100 rpm		19	13	18	11	18	14
	6 rpm		8	5	8	4	7	4
	3 rpm		7	5	8	4	7	4
	Gels 10"	lb/100ft <sup>2</sup>	8	5	8	4	6	4
	Gels 10'	lb/100ft <sup>2</sup>	8	5	8	4	7	5
	Plastic Viscosity	cP	21	18	19	20	23	27
	Yield Point	lb/100ft <sup>2</sup>	13	9	13	3	14	7
<b>Filtration:</b>	Temperature	°C	150	150	150	150	150	150
	HTHP Fluid Loss	mL oil	1	1	2	4	<1	<1
	Filter cake	mm	1	2	2	1	1	2
Measured Density		ppg	13.3	13.2	13.2	13.3	13.4	13.3
Electrical Stability		Volts	844	799	643	548	672	594
<b>Sag test</b>	Supernatant Liquid	mL		35		56		110
	Sag Factor			0,511		0,506		0,540

**Table 3: Properties of 13.3 ppg OBMs with Mn<sub>3</sub>O<sub>4</sub>, ilmenite and barite**

Chemicals	Mass in (gm)			Mixing time (min)
Mineral oil	313,3	313,3	313,3	
Amine based wetting agent	18	16	14	5
Fatty acid based emulsifier	14	14	14	5
Organophilic clay	1,6	1,6	3	10
Lime	16	16	16	5
Lignite based fluid loss agent	16,8	16,8	16,8	5
Polymeric fluid loss agent	7,2	7,2	7,2	5
Dispersant	2,4	2,4	2,4	5
CaCl <sub>2</sub>	31	31	31	15
Water	104,5	104,5	104,5	
Manganese Tetraoxide (Mn <sub>3</sub> O <sub>4</sub> )	1106			10
Ilmenite (FeTiO <sub>3</sub> )		1186		10
Barite (BaSO <sub>4</sub> )			1277	10

*Table 4: Recipe and mixing procedure of 17.2 ppg HPHT OBMs with Mn<sub>3</sub>O<sub>4</sub>, ilmenite and barite*

Test results			Mn <sub>3</sub> O <sub>4</sub>		Ilmenite		API Barite	
			BHR	AHR	BHR	AHR	BHR	AHR
Temperature °C		°C	150	150	150	150	150	150
Period static aging		Hours		16		16		16
<b>Rheology:</b> Temp °C			50	50	50	50	50	50
	600 rpm		82	82	97	114	124	122
	300 rpm		47	48	50	58	67	64
	200 rpm		35	34	36	40	47	43
	100 rpm		20	20	20	23	27	25
	6 rpm		5	5	3	4	6	5
	3 rpm		4	4	3	4	5	4
	Gels 10"	lb/100ft <sup>2</sup>	6	5	2	2	4	4
	Gels 10'	lb/100ft <sup>2</sup>	6	7	2	2	6	5
	Plastic Viscosity	cP	35	34	47	56	57	58
	Yield Point	lb/100ft <sup>2</sup>	12	14	3	2	10	6
<b>Filtration:</b>	Temperature	°C	150	150	150	150	150	150
	HTHP Fluid Loss	mL oil	2,8	2,2	10	5	1	1
	Filter cake	mm	1- 2	<1	7	6	<1	2
Measured Density		ppg		17.0	17.1	17.3	17.2	17.2
Electrical Stability		Volts	944	728	598	570	726	542
<b>Sag test</b>	Supernatant Liquid	mL		Ca 10		8		49
	Sag Factor			0,506		0,508		0,522

*Table 5: Properties of 17.2 ppg HPHT OBMs with Mn<sub>3</sub>O<sub>4</sub>, ilmenite and barite*

Chemicals	Mass in (gm)			Mixing time (min)
	g	g	g	
Water	320	320	320	0
Starch	4	4	4	2
KOH	0.75	0.75	0.75	5
Anionic cellulose	6	6	6	2
KCl	60	60	60	2
Acrylic dispersant	14	14	14	2
Mn <sub>3</sub> O <sub>4</sub>	850	-	-	5
FeTiO <sub>3</sub>	-	904	-	5
BaSO <sub>4</sub>	-	-	988	5

*Table 6: Composition of 19.2 ppg WBMs with Mn<sub>3</sub>O<sub>4</sub>, ilmenite and barite as weighting agents*

			Mn <sub>3</sub> O <sub>4</sub>		Ilmenite		Barite	
Temperature		°C		80 °C		80 °C		80 °C
Period static aged		Hours		16		16		16
Rheology			50 °C	50 °C	50 °C	50 °C	50 °C	50 °C
	600 rpm		74	57	102	92	134	112
	300 rpm		45	37	54	48	70	58
	200 rpm		33	28	41	34	49	40
	100 rpm		20	18	23	19	24	19
	6 rpm		6	7	4	4	2	2
	3 rpm		5	6	4	3	1	1
	Gels 10"	lb/100ft <sup>2</sup>	5	4	1	1	1	1
	Gels 10'	lb/100ft <sup>2</sup>	8	4	3	2	3	1
	Plastic Viscosity	cP	29	20	48	44	64	54
	Yield Point	lb/100ft <sup>2</sup>	16	17	6	4	6	4
Filtration	Temperature	°C	80	80	80	80	80	80
	HTHP Fluid Loss	mL water	15.6	9.2	10.8	10	8.4	5.6
	Filter cake	mm	7	4	1	2	-	<1
Sag test	Supernatant Liquid	mL		-		-		-
	Density - Top	g/cm <sup>3</sup>		2.37		2.32		1.92
	Density - Bottom	g/cm <sup>3</sup>		2.38		2.42		2.55
	Sag Factor			0.501		0.511		0.570
pH			9.2	9.8	9.2	9.4	9.8	9.2

*Table 7: Properties of 19.2 WBMs with Mn<sub>3</sub>O<sub>4</sub>, ilmenite and barite before and after static heat aging at 80°C*

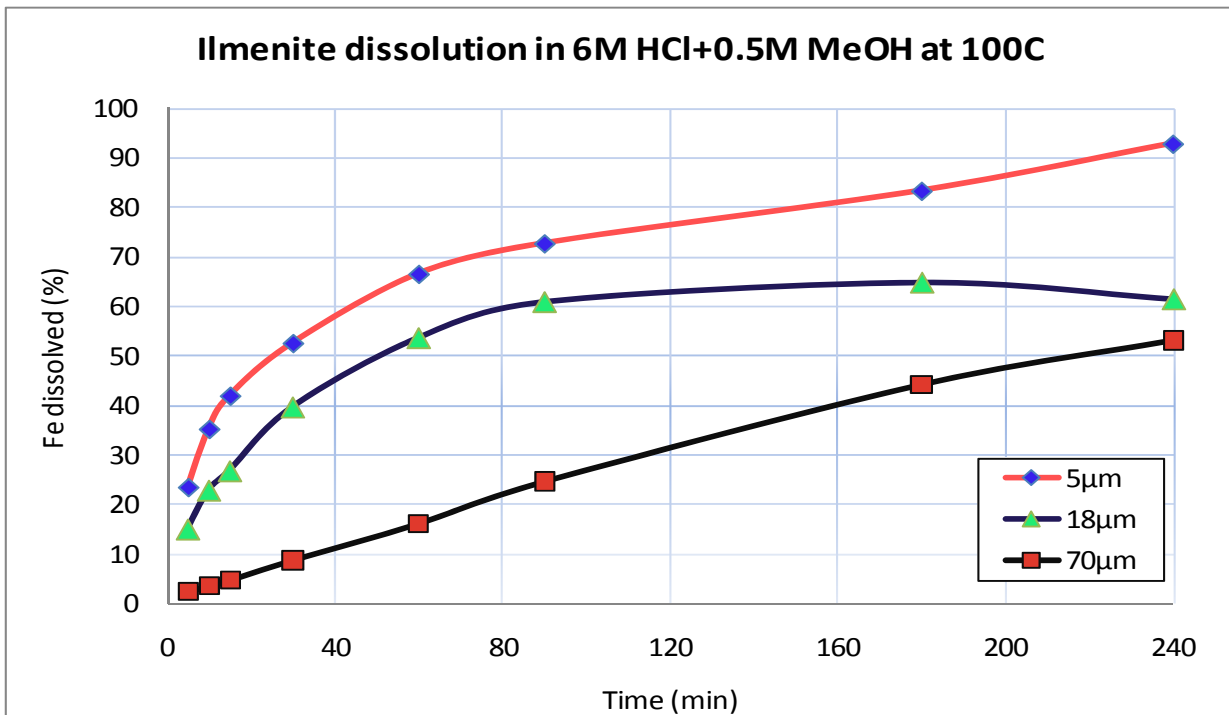


Figure 3: Acid solubility of ilmenite as function of particle size. The dissolved Fe in mg/l is at 180min at 100°C

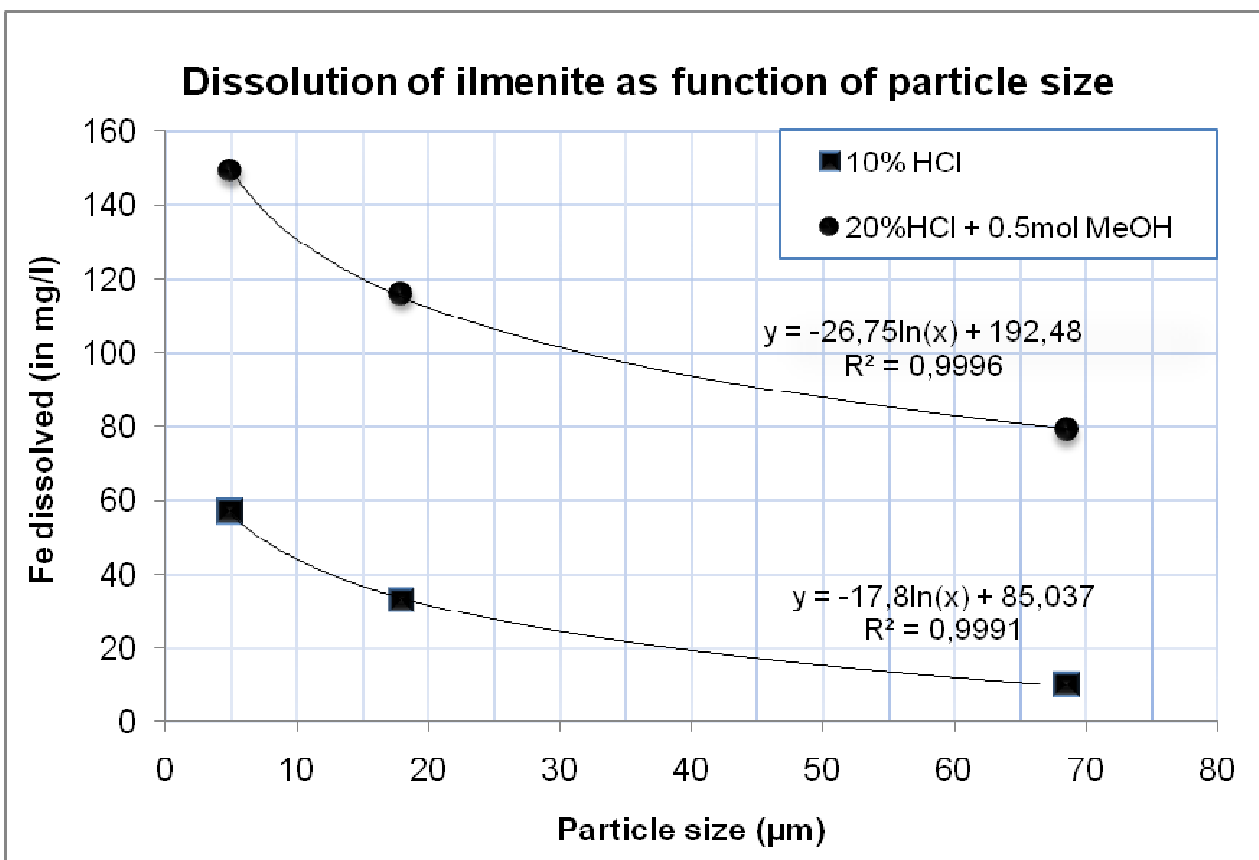


Figure 4: Acid solubility of ilmenite as function of particle size. Dissolved Fe in mg/l is at 180min at temp of 100°C