E-SY ALUMINAS – THE NEW SOLUTION FOR PUMPABILITY OF SILICA FREE HIGH PERFORMANCE TABULAR ALUMINA AND SPINEL CASTABLES

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ABSTRACT

Good pumpability of low water demand castables is a key technical challenge for high performance, high purity castables based on Tabular alumina and Spinel. Additions of microsilica to achieve the desired rheology for pumping at reasonable pressure in piston pumps used for pumpcasting and shotcreting is not an option since it would reduce the thermo-mechanical strength at high temperatures and reduce the corrosion resistance.

The challenge was to develop alumina matrix concepts, which give castables a low dilatancy even under extreme shear conditions as exists in double piston pumps. The products E-SY 1000 and 2000 are solutions to this challenge. Trials have proven that Tabular alumina castables using E-SY 1000 and E-SY 2000 as matrix aluminas fulfill the rheological requirements of these pumps.

The difference between E-SY 1000 and E-SY 2000 is their chemical composition. E-SY 1000 contains only alumina components and E-SY 2000 contains alumina and alumina-rich Spinel. It is well known in the industry that the addition of fine alumina-rich AR78 Spinel to castables will significantly increase both slag resistance and hot strength. E-SY 2000 combines superior pumpability with the advantages of alumina-rich Spinel. The use of E-SY 2000 will be of high interest, for example, in steel ladle applications.

The paper will discuss the properties and application results of the new aluminas based on laboratory tests and field trials.

INTRODUCTION

Synthetic alumina based raw materials are increasingly being used in a wide range of industries such as refractories, ceramics, and polishing. There is a trend in refractory industries towards monolithic linings to replace bricks. Common technologies pumping refractory castables during installation are different in Europe and North America. In Europe, mixer-pumps often are used, which pump castables after batch mixing by pressurized air through a hose. This technology is not very demanding regarding the castable rheology and either vibration or self-flowing castables can be used. In North America castables are also batch mixed. However, pumping is often done with double piston pumps, which create high shear rates within the castable. Due to the high shear rates, castables with very low dilatancy are required which demands special attention to the castable composition and rheology. Pumpability of castables is a must for shotcreting installation, but lower dilatancy is generally an advantage for easier installation. The pumpability over long distances, even with small low-power mortar pumps, is of interest e.g. for low volume repairs. In addition to pumping, low dilatant castables have advantages also in other applications, such as self-flowing installation.

The castable rheology is mainly defined by the matrix composition below 100 µm, and particularly below 45 µm, e.g. self-flowing castables require a higher amount of fines than vibration castables[1,2]. It is difficult to achieve pumpability of high purity Tabular and Spinel castables since very low dilatancy is required. Initial formulations used silica fume as superfine matrix components but refractoriness and thermo-mechanical stability decrease and therefore this limits the use in high performance applications[3].

WHAT MAKES A PUMPABLE MIX?

A high quality castable formulation is principally set up by a coarse and fine fraction[1]. The coarse fraction, which contains aggregates such as tabular alumina or magnesia alumina spinel is typically defined as the part of the formulation above 45 µm up to 6-10 mm comprising about 65-75 wt.-% of the total formulation. Since aggregates are rigid components, the flow behaviour of a castable is mainly influenced by viscous deformation during the flow process. The quantity of matrix fines, defined as the fraction below 45 µm, must be high enough to prevent collision of coarse aggregates during flow. They have an important influence on the rheology, workability and strength development of the final product. Lab results show that vibration castables require approx. 25% matrix fines compared to more than 30% for self-flow castables.

As a first approach, self-flow castables were used for pumping trials. However, the experience was that their rheology was too dilatant and that they could not be pumped at all. Detailed lab investigations showed that neither vibration nor self-flow castables had a rheology to achieve steadily also pumpable properties. The dilatancy of a castable has to be low enough to achieve pumpability, however, there is no direct link between flow behaviour and dilatancy.

This observation is also confirmed in the literature[4,5]. The theory that low mixing energy is required for pumping was tested with a special rheometer[6]. Pumping conditions were simulated by a special lid in the rheometer, which leads to volume restriction similar to pumping. Pileggi showed that self-flow is not the criteria to judge the pumpability of a castable. In contrary to the expectation, castables mentioned as 'easy mixing castables' showed self-flow or vibration rheology. Low mixing energy and torque values at high shear rates under volume restriction have to be achieved to ensure pumpability of a castable composition.

Based on the experience gathered by extended lab trials, the E-SY series has been developed as a solution to provide low dilatancy for high purity (silica free) Tabular and Spinel mixes. It is composed in such a way that addition of 20-30 % of E-SY to the formulation leads to pumping rheology of a castable.

As a qualitative evaluation of the pumpability of a mix, the so-called ball drop test has been developed. In the drop test, a steel ball (110 g, 30 mm diameter) falls from one meter above the mix surface into the mix and the time required from surface contact to complete submersion of the ball is measured. The measurement must be conducted in a vessel with a depth not less than three times greater than the steel sphere diameter. The result is given as time in seconds for complete submersion of the ball. By experience, a castable with a ball drop time of less than 3 seconds is considered to be suitable for pumping.

CHARACTERISTICS OF E-SY ALUMINAS

Two different E-SY products are available, which differ in mineralogical composition. E-SY 1000 is pure alumina based, whereas E-SY 2000 contains alumina and alumina rich spinel. Both are bimodal reactive aluminas, with typical d50 and BET of 1.9 µm and 3.3 m²/g (E-SY 1000) and 1.9 µm and 3.5 m²/g (E-SY 2000). Chemical and physical properties are summarized in **Table I,** a typical Particle Size Distribution (PSD) of E-SY 2000 is shown in **Figure 1** and of E-SY 1000 in **Figure 2**.

Table I: Typical data of E-SY 1000 and E-SY 2000

Chemical Analyses		**E-SY 1000**	**E-SY 2000**
Na_2O	%	0.3	0.1
MgO	%		12.0
SiO_2	%	0.10	0.10
Fe_2O_3	%	0.04	0.04
Physical Properties			
BET	m²/g	3.3	3.5
Cilas D50	µm	1.9	1.9
Cilas D90	µm	16	16
Grain size distribution		Bimodal	Bimodal

Figure 1: E-SY 2000

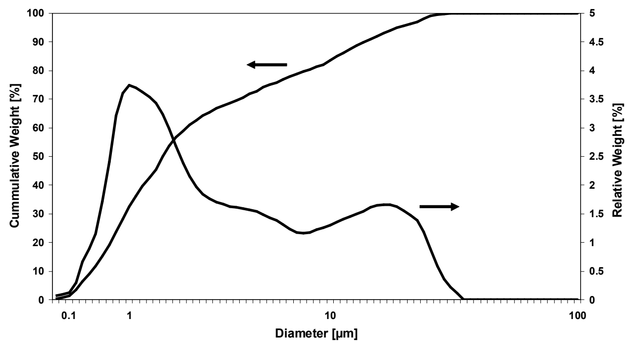
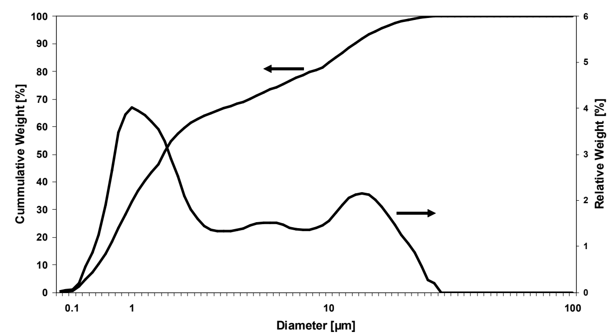


Figure 2: E-SY 1000



E-SY ALMINAS are, like other multi-modal reactive aluminas, produced by co-grinding that gives higher homogeneity and improved rheological behaviour compared to single component formulations. The advantage of co-grinding can also be seen in the results of the ball drop test, where E-SY gives an immersion time of 1 second but blended single reactive aluminas of similar composition is 3 seconds.

Table II: Composition & Properties of Pumpable Castables with Different Top-Cuts

Components \ Castable			Top 2 LPP	Top 1 LPP	Top 6 DPP	Top 2 DPP
			Low power pump		Double piston pump	
Coarse Fraction (0.5-6mm)	T-60/T-64	%	45	40	38	45
Fine fraction (0-0.5 mm)	T-60/T-64	%	30	30	30	25
Reactive Alumina	E-SY 2000	%	20	25	27	25
Cement	CA-270	%	5	5	5	5
Dispersing Aluminas	ADS 3	%	0.7	0.7	0.7	0.7
	ADW 1	%	0.5	0.5	0.5	0.5
Organic fibres	17 DTEC	%	0.1	0.1	0.1	0.1
Water		%	6.6	7.0	5.6	5.8
Ball drop		sec	1	2	2	1
Pressure max.	Low power pump	Bar	2	2	--	--
	Double pistol pump	Bar	--	--	170	170

CASTABLE PROPERTIES WITH E-SY 2000

Four low cement castables based on E-SY 2000 were developed for pumping trials in a *low power mortar pump* or a *double piston pump*. The low cement castables contain a range of coarse aggregate sizes (T-60/T-64), and up to 27 % E-SY 2000 and dispersing additives (ADS 3, ADW 1). These castables are described in **Table II**.

Depending on the castable composition, low dilatancy is achieved with 5.6 to 7.0 % water, which is reflected in a ball drop of 1-2 seconds. From all castables flow properties were determined with a cone test (lower ∅ 100 mm, upper ∅ 70 mm, height 80 mm). The castable setting time has been measured by the EXO method, recording the temperature development during setting. Test pieces have been prepared and tested according to the European standard ENV 1402 "Unshaped refractory products", Part 5 and Part 6. Hot properties (HMOR), density water absorption and open porosity were measured by DIFK, Bonn, Germany. Further details on sample preparation are described in a previous paper[1].

For trials in a *low **p**ower mortar **p**ump*, castables Top 2 LPP and Top 1 LPP were used. Top 2 LPP contains coarse aggregates up to 2 mm and has a low dilatancy with 20 % E-SY 2000 and 6.6 % water. The rheology of the castable with a top cut of 1 mm is similar when 25 % E-SY 2000 and 7 % water are used (TOP 1 LPP). Both castables have a flow at 30 minutes (F30) of >280 mm and a ball drop time of 1–2 seconds. The EXO start was adjusted to 4 hours to avoid setting and clogging in the hose during the trials. However, the setting time can be adjusted by changing the ADS 3/ADW 1 ratio. Despite the relatively high water content of the castables, test pieces show technically sufficient strength in all temperature ranges, e.g. CCS >30 MPa (1000 °C) and >165 MPa (1500 °C) and HMOR at 1500 °C between 12 and 16 MPa. The bulk density is around 3 g/cm³ and there is little shrinkage at 1500 °C (permanent linear change: max -0.33 %)

PUMPING TRIALS - MORTAR PUMP

Based on the lab results, both LPP castables were tested with a simple low power mortar pump (5 EVTM Pump, Putzmeister Mörtelmaschinen GmbH). Pump trials have been performed with 70 kg castable mix (coarse and fine Tabular sizes, Reactive Aluminas, Calcium Aluminate Cement, fibres, and Dispersing Aluminas). A mix of E-SY 2000 and dispersing aluminas was filled into the mixer and homogenized with the whole calculated quantity of water for three minutes. Afterwards the tabular sizes and the cement where added, and mixing was continued for another six minutes. This procedure was required since the engine power of the mixer was initially not sufficient to homogenize the castable component in one step. Once the castables were homogenized, they could both be easily pumped through a hose with 50 mm diameter over a height of three meters. During pumping, the pressure never exceeded 2 bar. Due to the limited distance between the screw and the casing of mortar pump a maximum top cut of 2 mm was chosen to avoid clogging by coarser grains.

Table III: Characterisation of pumpable castables

	Castable	**Top 2 LPM**	**Top 1 LPM**	**Top 6 DPP**	**Top 2 DPP**
	Pretreatment	Low power pump		Double piston pump	
Flow [mm]	10 min	281	307	247	239
	30 min	279	308	249	232
	60 min	258	299	247	235
EXO	Start 1 / min	578	281	254	413
	Start 2 / h	13.3	7.2	7.3	10.6
	Exo Max / h	16.9	9.9	9.5	13.0
CMOR [MPa]	20°C / 24h	3	2	3	3
	110°C / 24h	12	10	13	14
	400°C / 5h	8	8	12	12
	1000°C / 5h	12	9	12	14
	1500°C / 5h	48	56	59	25
CCS [MPa]	20°C / 24h	10	11	13	14
	110°C / 24h	54	47	59	55
	400°C / 5h	44	37	63	67
	1000°C / 5h	39	29	52	51
	1500°C / 5h	168	165	309	238
Permanent Linear Change [%]	110°C / 24h	±0	±0	±0	±0
	400°C / 5h	-0.08	-0-09	-0.06	-0.06
	1000°C / 5h	-0.03	-0.06	-0.03	-0.04
	1500°C / 5h	-0.03	-0.33	-0.32	-0.37
HMOR [N/mm^2]	1500°C / 5h	16	12	22	22
Bulk Density [g/cm³]	110°C / 24h	3.06	3.00	3.01	3.02
	1000°C / 5h	3.00	2.88	3.04	3.03
	1500°C / 5h	3.01	2.98	3.02	3.04
Apparent Porosity vol.-%	110°C / 24h	16.1	17.7	13.7	15.3
	1000°C / 5h	19.0	22.9	18.1	18.3
	1500°C / 5h	19.5	20.6	18.9	18.5
Water absorption [wt-%]	110°C / 24h	5.3	5.9	4.4	5.1
	1000°C / 5h	6.3	7.9	5.9	6.1
	1500°C / 5h	6.5	6.9	6.2	6.1

PUMPING TRIALS - DOUBLE PISTON PUMP

For the pump trials with a **d**ouble **p**iston **p**ump, castable Top 6 DPP with a maximum grain size of 6 mm was set-up. With 27 % E-SY 2000 and only 5.6 % water pumpable consistency could be achieved. In addition, the Top 2 LPP was slightly adjusted to achieve low dilatancy with only 5.8 % water and 25 % E-SY (Top 2 DPP). Both castables have a F30 of >240 mm and a ball drop time of 1-2 seconds. The pump trials were conducted in a double piston pump MBS 70/80 (Montanbuero GmbH). All components of the castable were dry-mixed before the pump trial. Castable batches of 500 kg were mixed with water, transferred to the pump vessel and a further 250-500 kg were mixed to complete the required quantity. Despite the high shear rates in the double piston pump, both castables could be easily pumped over a distance of 50 m through several rubber hoses and steel pipes with 50 mm and 38 mm diameter. The pumping pressure never exceeded 170 bar during the whole pump trial. The dilatancy of both castables is considered to be low enough for pumping even over longer distances, which is considered to be a normal range by the experienced operators.

Due to the better mixing equipment attached to the double piston pump, castables used for the trial required less water than using the low power mortar pump and easy pumpability was achieved with only 5.6 % water. The lower water content results in higher strength of the castable samples compared to samples from the mortar pump trial. E.g. the CCS at 1500 °C raises from 165 MPa (Top 1 LPP) to more than 300 MPa (Top 6 DPP) and the HMOR rises to 22 MPa.

All castables and pump trials have been performed with the Spinel containing E-SY 2000. However, they could also have been done with the high alumina based E-SY 1000 as the castable rheology is equivalent with the 2 materials.

CONCLUSIONS AND OUTLOOK

The trend toward monolithic lining requires state of the art installation methods and appropriate castables formulations. The dilatancy of high purity vibration and self-flowing castables limits the use of pumping technology, especially when double piston pumps are used. Early pumpable formulations used silica fume for the matrix superfines to reduce dilatancy but it also caused a negative effect on refractoriness and thermo-mechanical strength. With the E-SY 1000 (pure alumina) and E-SY 2000 (spinel containing) two types of high purity Reactive Aluminas are available which enable the design of low dilatancy castable formulations. Several castables compositions based on E-SY 2000 presented here gave reduced dilatancy compared to previous self-flowing castables. Field trials showed that E-SY castables are pumpable both with simple low power mortar pumps and with double piston pumps, which are very demanding with respect to the castable rheology. Castables were easily pumped over a distance of 50 m though hoses with diameters of 38 and 50 mm.

Although not completed in time for paper submission additional work is underway to further improve the rheology of the formulations for shotcreting. In other work E-SY 1000 and E-SY 2000 have shown themselves to be excellent sources of sub-micron alumina fines for vibration and self-flow castables. The rheology with the E-SY materials is softer than with traditional reactives that are designed to optimise particle packing.

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