SUITABILITY OF THE SULCIS COAL FOR CWS PREPARATION

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ABSTRACT:

A ***Coal Water Slurry*** (CWS) containing about 65-70% solids by weight can be defined as a stable combustible mixture having a high heat power, in spite of the presence of water. A suitable particle size distribution accompanied by a small quantity of additives contribute to the obtainment of such properties enabling handling, trasportation, storage and combustion as a heavy fuel oil.

The utilization of CWS offers a number of economic and environmental advantages concerning either the transport and storage operations (better management of coal stockpiles, lower intensity of traffic ….) and the final combustion (CWS can be the starting phase for the application of a number of clean coal technologies including gasification and hydrogen production)

Aim of the research work dealt with in the paper is the study of the influence on suspension stability and rheologic characteristics (viscosity, thyxotropy) of various parameters such as:

- Particle size distribution
- Type and dosage of a fluidising additive
- Type and dosage of a stabilising agent
- Proportion of coal in the mixture.

Optimum CWS should be characterised by a high heat value, good stability and a viscosity low enough for pipeline delivery.

Results obtained through a systematic experimental programme, while confirming a better aptness of high.rank coals, have shown that also the sub-bitominous coal mined in the Sulcis coalfield is amenable to the preparation of CWS to be burned in the nearby power stations.

1. FOREWORD

Coal is yet again one of the most important sources of energy, since it covers about 26% of the world demand and generates up to 41% of electricity.[1], with a contribution likely to raise in the future.

In Italy too, in spite of an energy mix strongly unbalanced towards the use of natural gas, the employment of steam coal is growing, also because some oil-fired power stations are being converted to coal, while 13 already existing coal-fired plants are being restructured.

The future of coal can be summarized by the sentence agreed upon by the most important Countries: *Coal is an abundant resource in the world. It is imperative that we figure out a way to use coal as cleanly as possible* [2],

In compliance with this, the development of clean technologies for coal has been progressively promoted in the last years under the pressure of environment protection issues, attaining some important technical results in terms of pollution control, with the goal of reducing sulphur dioxide emissions by at least 80%, particulate matter by 75 % and nitrogen oxides by 60% while recovering 100 % of ash.

Following this innovation progress, some important targets have been hit by the Italian power stations, witnessed by a 45% energy recovery from coal, compared with a European mean level of 39%. Since the last studies by the International Agency for Energy predict the attainment in Europe of an average efficiency of 42% by the year 2020, it can be said that Italy is 20 years ahead in the pursue of this goal.

Among the Clean Coal Technologies (CCT), the Coal Water Slurry technology (CWS) can play an important role, owing to a number of advantages such as: the possibility of storing this kind of fuel without the problems posed by the management of coal storage heaps, the opportunity of delivering the coal over long distances via pipeline without the environmental impacts posed by airborne dust and by heavy traffic, and the chance to replace fuel oil in the power stations with minor technical adjustments.

A CWS is a complex system where a number of parameters are involved concerning the definition of rheologic and flow characteristics of the mixture.

The present work was aimed at assessing the feasibility of preparing CWS with the Sulcis coal, mined in the Nuraxi Figus district in Sardinia (Italy), classified as sub-bituminous with high sulphur content and thus considered unsuitable for that particular use.

In the study, carried out at the DIGITA laboratories of the University of Cagliari in the frame of a co-operation project with ENEA, the National Agency for Energy and Environment, the results obtained with the “Sulcis” are compared with those obtained with a high-rank coal imported from Russia, used by the company Energy Coal for the preparation of CWS in its demonstration plant located in the industrial area of Oristano.

The performance of CWS obtained with each of the two kinds of coal was investigated as a function of particle size distribution, kind and dosage of fluidizing and stabilizing agents.

[1] *World Coal Institute 2008*
[2] *Energy Senate Confirmation Hearing January 13, 2009*

2. EXPERIMENTAL PLAN

2.1 Coal samples

The main characteristics of the two coals used for the tests are summarized in the following tables 1 and 2.

Table 1. Characteristics of the Sulcis coal determined with the LECO MAC 400 apparatus.

	AS SUCH [%]	DRY [%]
Moisture	8,07	
Volatile matter	47,59	52,12
Ash	7,87	8,56
Fixed carbon	36.46	39.66
Volumic mass [kg/dm3]		1.4285
Higher heat value [kcal/kg]		6,848

Table 2. Characteristics of the Russian coal made available by Energy Coal.

PARAMETERS	VALUES	METHOD
Total moisture [%]	7,65	ASTM 3302-89
Surface moisture [%]	1,32	ASTM 2013-86
Inherent moisture [%]	6,41	ASTM 3173-88
Volatile matter [%]	30,55	ASTM 3175-89
Ash [%]	12,97	ASTM 3174-89
Fixed carbon [%]	50,07	ASTM 3172-84
Total carbon [%]	70,12	ASTM 3178-84
Hydrogen [%]	4,08	ASTM 3179-84
Nitrogen [%]	3,74	ASTM 3178-84
Total sulphur [%]	0,26	ASTM 4239-85
Oxygen (diff.) [%]	8,83	ASTM 3176-84
Higher heat value [kcal/kg]	6.566	ASTM 3286-85
Lower heat value [kcal/kg]	6.321	ASTM 3286-85

The heat value of the Sulcis coal (hand sorted sample) appears higher that that of the Russian coal (run-of-mine sample) due to a small content of ash-forming mineral matter.

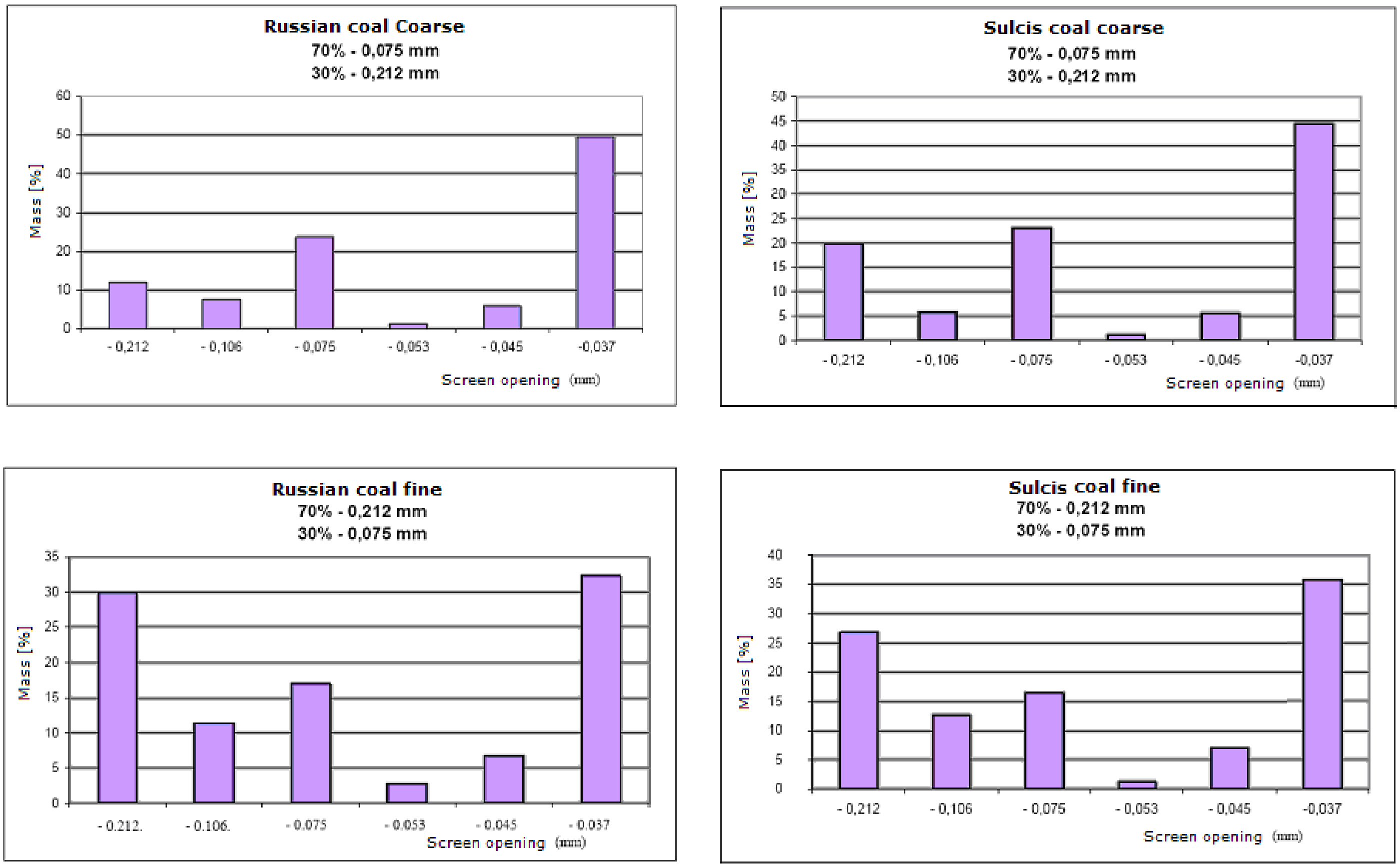
2.2.Comminution

Each coal sample was crushed in a jaw crusher and then dry ground in a rod mill in closed circuit with a control screen of 0.212 mm opening. Half of this product was further ground in similar way below 0.075 mm.

The feed samples, “coarse” and “fine”, for CWS tests were obtained by blending in two different proportions (70:30 and 30:70, respectively) the two products of comminution. The corresponding particle size distribution are reported in figure 1.

The histograms show a marked “bimodality” of the size distributions in compliance with the suggestions found in the literature in order to ensure better stability of the suspension with acceptable fluidity, taking also into account the energy content of the resulting CWS.

Figure 1. Particle size distribution of the feed material for CWS preparation



2.2 Additives

Basically two kinds of chemicals are used in the preparation of CWS: a fluidizing agent aiming at reducing the viscosity of the slurry for easier flow and injection into the boiler and a stabilizing agent for preventing settling along the delivery pipes and inside the storage reservoirs.

The additives should be selected in order to:

• maximize the coal load (yield) of the slurry

• provide better conditions for supply, handling and storage of CWS

• meet the requirements for safety, health and environment

• achieve economic advantages in the use of the fuel.

The choice of the chemicals for the tests has been based on the findings of scientific research as well as on some considerations concerning their market price and availability.

Following the above criteria, four agents have been selected:

– *Na–Esametaphosphate*, a dispersing agent widely used in froth flotation for reducing the viscosity of dense pulps;

– *Superwater®.* a polyacrylamide soluble polymer that proved the best as a result of a broad study carried out at the DIGITA's Waterjet Laboratories in the frame of a European Project aimed at improving the performance of abrasive suspension jets.

– *Proxanol®*, a copolymer of polythene and polypropylene ether behaving as a non-ionic surface active agent, used also in the medical field as softener and in general for providing "plasticity" to a variety of organic substances;

– *Rhodopol®,* a polysaccharide-based substance having low viscosity, often supplied in the form of concentrated aqueous solution prepared using specific water-soluble anionic copolymers.

The last two are currently employed in the preparation of CWS at the above mentioned Energy Coal demonstration plant.

For the experimental tests each pair of chemicals (a stabilizer + a fluidizer) were added to the coal suspension in the form of solution in distilled water as follows:

• Combination A (Proxanol + Rhodopol)

• Combination B (Na–Esametaphosphate + Superwater).

The additives were prepared in the form of solutions in distilled water that were dosed in the slurry in variable quantities with respect to coal as shown in table 3.

Table 3. Dosage of additives in the CWS as % of their solutions with respect to coal and in grams of dry substance per tonne of coal .

Fluidizer				Stabilizer			
Proxanol (0,3%)		Metaphosphate (1%)		Rhodopol (0,02%)		Superwater (0,3%)	
%	*g/t*	*%*	*g/t*	*%*	*g/t*	*%*	*g/t*
0.65	455	2.00	200	4.00	8.00	0.27	8.1
0.96	672	3.06	306	6.04	12.08	0.42	12.6
1.27	889	4.19	419	8.19	16.38	0.58	17.4
1.61	1127	6.60	660	10.44	20.88	0.90	27.0
2.37	1659	11.55	1155	15.06	30.12	1.56	46.8

2.3 Water

In the experimental study distilled water in proportions variable from 37% to 50% was used for the coal suspensions. Since the importance of water quality for the performance of CWS is well known, the influence of the chemical composition of the water will be studied in a further development of the research.

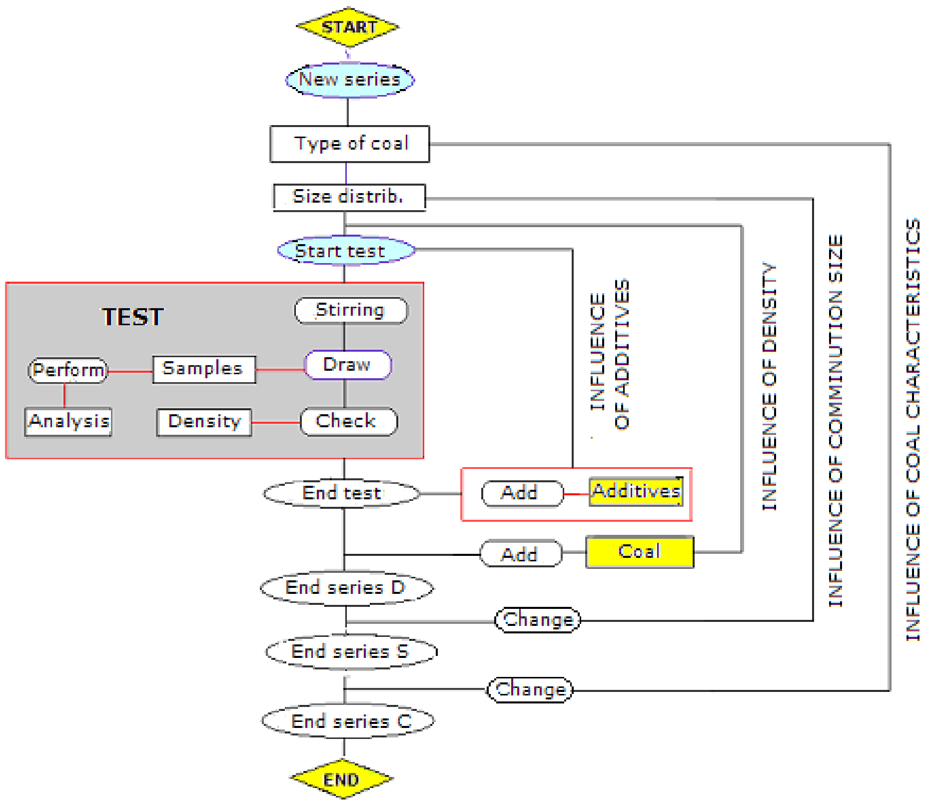
2.4 Experimental procedure

The experimental plan was conceived in order to study the features of CWS as a function of coal properties (reflected by its rank), with special reference to the Sulcis coal under variable experimental conditions. In particular the following aspects have been explored:

- the particle size distribution
- the water-to-coal ratio in the CWS
- the type and dosage of a fluidizing agent
- the type and dosage of a stabilizing agent
- the stirring time.

The structure of the testing plan is sketched in the block diagram of figure 2.

Figure 2. Structure of the testing plan



According to it, the influence of additives was investigated firstly at low density of the suspension. Then the density was increased by steps (each corresponding to a 5% increase of the proportion of coal in the mixture). Each series of tests was performed by simply increasing the additive dosage or adding new coal to the initial suspension, weighting 2 kg and having a coal proportion of 50%. On completion of each series, the procedure was repeated for a new sample of coal (different in type and size distribution).

At the end of every standard test, after about one hour stirring time, three samples were taken by means of a 50 cc syringe: one for the determination of sedimentation parameters, one for the measurement of rheologic properties and the third for checking purposes. Therefore for each of the four series of tests 10 triplets of samples were obtained:

- 4 for the tests with increasing additive dosage at constant density
- 4 for the tests with increasing density while keeping the additive concentration at its maximum value
- 2 for the control tests with decreasing density (backwards path) again at constant peak additive concentration

After each sampling operation the density of the mixture was checked and, whenever necessary, a small quantity of water was added for compensating the evaporation losses.

The three components of the system (water, coal and additives) were intimately mixed inside a stirring vessel having a capacity of 6 litres provided with a rotor fitting a vertical shaft to which a counter-rotating revolution motion is also applied by means of a cam (figure 3). Rotation velocity can be varied continuously.

The following data have been obtained and processed:

• settling curves for the evaluation of CWS stability

• viscosity curves

• thixotropy values

Moreover, some considerations have been drawn concerning the heat value of the CWS.

Figure 3. Mixing vessel

Figure 4. Viscometer

2.5 Data obtained

2.5.2 Viscosity curves

Rheologic parameters (viscosity and thixotropy) have been measured by means of a *HAAKE RotoVisco 20* viscometer using the *RheoWin 3* Software (figure 4).

Viscosity has been obtained through the measurement of the tangential force per unit surface [Pa] as a function of the velocity gradient determined by the speed of the viscometer's rotor from 5 to 200 s^{-1} over a total duration of the operation of 60 s.

The asymptotic value of the curve has been assumed as representative of the fluidity of the mixture.

A typical curve of tangential tension and corresponding viscosity is shown in figure 5.

Figure 5 Curves of tangential tension and viscosity as a function of time with linearly increasing velocity gradient.

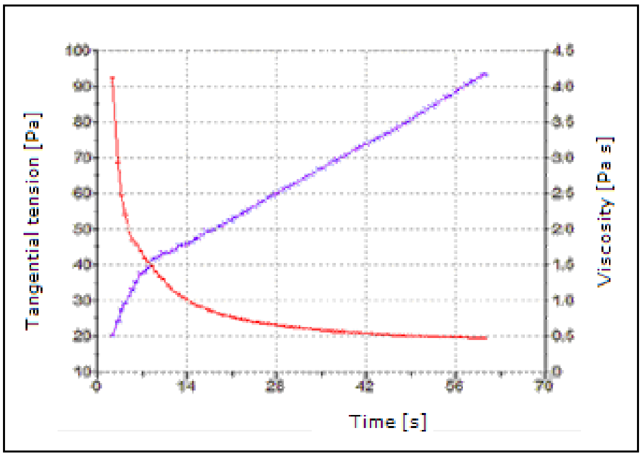
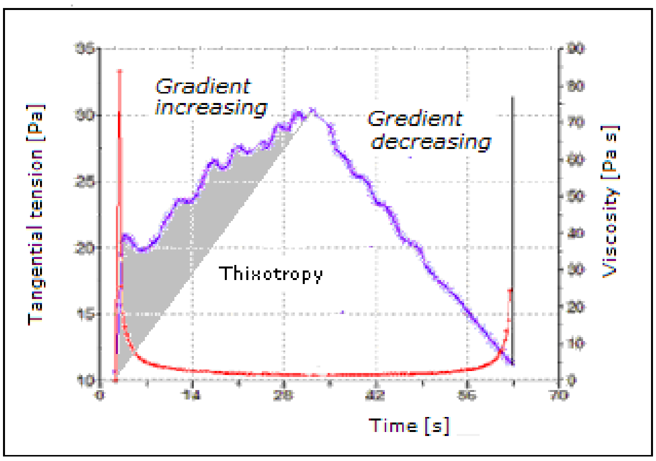


Figure 6 Curves of tangential tension and viscosity as a function of time after a complete up/down cycle of he velocity gradient.for thixotropy assessment



2.5.3 Determination of thixotropic characteristics

Thixotropy can be defined as the capability of non-Newtonian fluids (pseudoplastic, Bingham, ….) to modify their viscosity when subject to shear actions or in the case of progressive agitation starting from quiet conditions.

Under these circumstances a slurry can pass from a thick, almost solidified state to that of a fluid or, more generally, from gel to liquid. Therefore thixotropy reveals the inertia of a still suspension to flow freely when forced to move.

Thixotropy has been determined by detecting the difference in the shear resistance in the two cases of increasing or decreasing velocity gradient *dV/dr* as a function of time, where *V* is the peripheral velocity of the viscometer's rotor and *r* is the radial distance from the rotor's rim.

A measure of thixotropy is represented by the difference between the areas below each of the two branches of the curve, both over a base interval of 30 s.

A typical curve giving the trend of shear resistance allowing the determination of thixotropy is shown in figure 6 together with viscosity as a function of time.

2.5.1 Sedimentation curves

The stability conditions of CWS have been assessed through the observation of the displacement as a function of time of the meniscus separating the layer of clear water from the underlying settling suspension inside a graduated cylinder (figure 7).

Settling velocity can be represented by the parameter t_{80} i.e. the time necessary for achieving 80% of the final volume of clear water in the sedimentation test. (figure 8).

Figure 7. Graduated cylinder for settling assessment

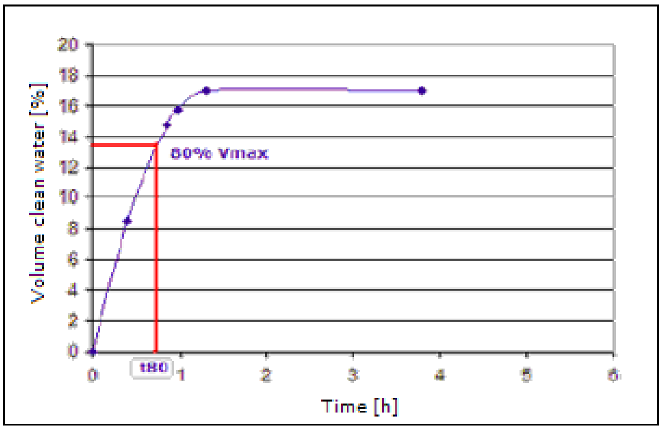


Figure 8. Typical sedimentation curve showing the ***t_{80}*** parameter

3. RESULTS AND DISCUSSION

The observed values and tendencies allow to draw with enough clearness and reliability the most important considerations useful for the preparation of coal-water slurries.
Based on experimental data the following aspects concerning the yield, as well as the stability and rheologic characteristics of the mixture under the simplified assumption of independency of the variables and parameters can be highlighted.

3.1 Influence of coal properties

Generally speaking, the results obtained with the Sulcis" (younger, porous, oxidized, rich in volatile matters) compared to those with the Russian coal (higher in rank), put into evidence a better suitability of this latter, at least as far as stability is concerned, in agreement with the knowledge reported in the literature, in spite of its relatively high ash content, being it an untreated raw material (above 12% against the 7% of the hand picked Sulcis coal, actually averaging about 20% after gravity concentration of the run-of-mine).

Table 3. Influence of size distribution and kind of additives on CWS stability for the two coals tested (lower values are for the higher additive dosage).

	FINAL VOLUME OF CLEAR WATER [%]			
Additives	*Proxanol + Rhodopol*		*Na-metaphosphate + Superwater*	
Size Distribution	*Coarse*	*Fine*	*Coarse*	*Fine*
Coal				
SULCIS	15 - 18	15 - 16	17 - 19	16 - 25
RUSSIAN	16 - 18	8 - 12	13 - 15	11 - 16

In fact:

• the final volume of clear water in sedimentation tests is significantly lower for the Russian coal, as represented in table 3 giving the results at low density (coal content 50%) with varying kind and dosage of additives. Moreover the behaviour of the two coals depends on particle size distribution;

• concerning the parameter $\mathbf{t_{80}}$ that describes concisely the kinetics of the settling process, it can be said that sedimentation is significantly slower in the case of the Russian coal for all the experimental conditions (table 4). Moreover sedimentation time is generally shorter in the case of coarser size distribution of suspended particles, as expected.

Therefore, in agreement with the suggestions of the technical literature, the mixtures will be more stable if high-rank coals are used.

• the viscosity of the slurry does not appear significantly influenced by the rank of the coal, at least in the range of low densities where it nears a level around 50 mPas for coarser size distributions and a 30 mPas for finer ones with both kinds of coal tested.

Table 4. *Influence of size distribution and kind of additives on settling time for the two coals tested*

	SEDIMENTATION TIME t_{80} [h]			
Additives	*Proxanol + Rhodopol*		*Na-metaphosphate + Superwater*	
Size Distribution	*Coarse*	*Fine*	*Coarse*	*Fine*
Coal				
SULCIS	0.4 - 0.7	0.5 - 1.5	0.2 - 0.7	0.6 - 1.5
RUSSIAN	1.0 - 1.8	1.6 - 3.5	1.5 - 2.2	1.5 - 3.5

• similar considerations hold also for thixotropy: there is not any substantial difference in rheologic behaviour between the Sardinian and the Russian coals, since the average values for this parameter are basically the same at equal particle size distribution.

It is trivial to remind that stability features of industrial CWS can be improved by means of a moderate stirring of the slurry inside the storage vessels.

3.2 Influence of slurry density

As the density of the mixture is increased by adding new coal, the final volume of clear water above the settling suspension diminishes. The waiting time for the attainment of stationary conditions varied from case to case as witnessed by the parameter t_{80} but after about 6 hours an asymptotic level was closely approached and the suspension remained practically stable in all cases.

In the tests with the combination of additives A (Proxanol + Rhodopol), starting from an initial value around 16-18% for the 50:50 mixtures, the final volume of clear water drops down to 2-4% (the higher value is for the coarser size distribution) in correspondence to the top density (about 63% of coal in the slurry). An inexplicable deviation from his rule has been observed for the Russian coal that exhibited a substantially stable performance (6-8%) even at low densities.

A similar behaviour is registered also with the additive combination B (Metaphosphate + Superwater), although in presence of some sporadic divergence from the decreasing trend, probably due to unknown experimental anomalies.

Concerning the sedimentation kinetics, the experimental curves put into evidence the presence of a minimum value for the parameter t_{80} corresponding to an intermediate density (around 55% coal), generally lower for the coarser size distributions. A diverging behaviour from the general trend, difficult-to explain, was exhibited by the Russian coal “fine” with additive combination A and by the Sulcis coal “coarse” with additive combination B.

Particularly important, for practical purposes, are the curves representing the viscosity of the slurry as a function of its density since they allow to single out the peak yield achievable for a given coal corresponding to a viscosity limit of about 1000 mPas, beyond which the fluid consistency is rapidly lost, making it impossible to handle the slurry as a pumpable liquid.

Such limiting viscosity is reached with a yield different for the two kinds of coal: slightly higher for the “Sulcis” with respect to the Russian coal (a surprising outcome!). The extrapolation of the curves to 1000 mPas show also that the yield is lower for the finer size distribution (according to expectation).

The better yield for the “Sulcis” could be explained by the lower ash content of the sample used for the tests as pointed out earlier.

The curves at maximum additive dosage for the determination of thixotropy as a function of density reflect strictly those for viscosity and they almost overlap each other except for a different scale.

3.3 Influence of coal comminution

Stability parameters are strongly influenced by the fineness of solids in the slurry in a sense that mixtures composed by coarsely ground coal are less stable and settle more quickly, the other conditions being the same.

This fact is substantiated by the data reported in Tables 3 and 4 concerning the influence of coal characteristics.

Also rheologic parameters of the slurry are affected by particle size distribution, given that the limiting value for viscosity as density increases is reached earlier in the case of finer comminution.

Therefore it is worthless to force too much the grinding operation, since the advantage of a better stability are outbalanced by a sharp deterioration of fluidity and thence by a lower yield of the mixture, besides the increase of CWS preparation cost. The use of additives can be helpful in the attempt to improve the performance, although being unable to reverse the trend.

3.4 Influence of chemical additives.

The influence of additives has been firstly studied in conditions of dilute suspensions (coal proportion: 50%) in the attempt to isolate the results from the concurrent influence of density and size distribution that could have “masked” the effect of additives.

The following aspects can be underlined concerning the stability features of the suspension:

- the final volume of clear water does not appear to depend considerably on the concentration of additives within the range studied: actually the curves obtained are almost flat and the expected reduction in solids/liquid segregation did not take place as dosage was increased.
- the trend of the kinetics parameter t_{80} as a function of additive dosage resulted to be poorly consistent for the different experimental conditions, thus providing no contribution to a better understanding of the mechanism of additive action.

• However if the attention is concentrated on the average values it emerges that the combination of additives A (Proxanol + Rhodopol) is slightly more advantageous than combination B (Metaphosphate + Superwater) for which sedimentation time is somewhat longer as already shown in Table 4.

No significant contribution to a clear comprehension of the effect of additives was obtained by the consideration of the rheology parameters in conditions of dilute suspension. In fact:

• the curves of viscosity as a function of additive dosage are almost flat with episodic and little significant deviations. However viscosity seems to be influenced by the different combination of additives as shown in table 5 giving the span of variation for viscosity within the range of additive concentration explored: the pair “Proxanol-Rhodopol” seems always more efficient in the control of viscosity than the pair “Metaphosphate-Superwater”.

Table 5. Influence of size distribution and kind of additives on the CWS viscosity

	VISCOSITY [mPa s]			
Additives	*Proxanol + Rhodopol*		*Na-metaphosphate + Superwater*	
Size Distribution	*Coarse*	*Fine*	*Coarse*	*Fine*
Coal				
SULCIS	25 - 30	40 - 50	30 - 32	55 – 65
RUSSIAN	35 - 40	45 - 55	36 - 40	58 - 62

• •rather different considerations can be put forward concerning thixotropy for which the curves exhibit the presence of a minimum value corresponding to the intermediate dosage of additives, followed by an increase at higher dosages; this behaviour is observed for both kinds of additives and for both types of coal as shown in table 6 where the increment of thixotropy starting from the minimum value is reported.

• however, while the maximum value is always observed for the higher dosage of additives, the minimum point shifts rightwards for the Russian coal with respect to the “Sulcis”. The general trend of thixotropy against additive dosage seems to indicate a certain interaction between the fluidizing and the stabilizing agent (the effect of the first prevails at the lower concentrations of additives whereas the second overcomes at higher dosages).

The fact that thixotropy increases while viscosity remains constant is certainly a positive aspect for the preparation and utilization of CWS.

Table 6. Influence of particle size and kind of additives on CWS thixotropy (into brackets the relative increment with respect to the minimum value)

	THIXOTROPY [Pa s] min - Max			
Additives	*Proxanol + Rhodopol*		*Na-metaphosphate + Superwater*	
Size Distribution	*Coarse*	*Fine*	*Coarse*	*Fine*
Coal				
SULCIS	3.2 – 7.3 *(1.28)*	5.2 – 6.5 (0.25)	1.1 – 8.6 (6,19)	4.2 – 10.5 (1.5)
RUSSIAN	3.5 – 8.0 *(0.45)*	8.0 – 9.7 (0.21)	6.0 – 10.5 (0.75)	8.2 – 16.6 (1.0)

The table shows also that the increment in thixotropy is higher for the coarsely ground Sulcis coal which derives benefits from the addition of the additives, especially “Superwater”, gaining a substantial improvement in performance, albeit starting from a lower minimum value with respect to the Russian sample, that however always holds higher thixotropy values.

3.5 Effect of mixing time

In the course of each series of experimental tests it emerged that:

- working at low density, the addition of a fluidizing agent had a poor effect on viscosity that remained practically constant while the dosage was increased
- working at high density, the sampling operation with the syringe became more and more difficult as the cumulative stirring time elapsed.

It was retained likely that a prolonged stirring could have caused a progressive shifting of particle size distribution towards the fine range, thus increasing the viscosity of the slurry and upsetting the effect of the additive.

In order to prove the correctness of this assumption, a series of long-duration tests has been carried out on a medium density mixture (60% solids) with the “coarse” sample of Sulcis coal in absence of additives.

It came out that a prolonged stirring time even exceeding 10 hours did not produce any significant change of CWS rheology. The observed variations are very small and can be due to possible changes of experimental conditions (fluctuation of temperature, release of ions in the solution, ….).

Also the variations in the final volume of clear water did not point out the onset of any phenomenon in some way imputable to the time (and energy) of mixing.

In order to cast some light on this aspect, two samples were taken and analysed, at the beginning and after 11 hours of continuous stirring. Wet screening at 53 μm gave the results reported in table 7.

Table 7. Variation of particle size after 12 h stirring time for a sample of Sulcis coal.

Size classes	Mass of size fraction [%]	
	Initial sample	Final sample
+ 0.053 mm	56.86	53.04
- 0.053 mm	43.14	46.96
Total	100.00	100.00

The small difference in weight between oversize and undersize does not fully elucidate the problem. Therefore it can reasonably be assumed that the observed results might be due also to a certain inertia in the interaction process between the additives and the suspension. It is also likely that some slow-evolution phenomena may occur, resulting in the liberation of inorganic or organic substances present in the coal and subsequent modification of the chemistry of the solution.

4. CONCLUSIONS

Experimental results show that it is possible to produce good CWS with the Sulcis coal, up to now considered unsuitable for this application.

However additional research efforts must be done aiming at further increasing the yield of the slurry and thus improving the economic feasibility of the operation.

The data obtained represent a sound starting base for the development of the technology through:

- a broader choice of additives in order to single out the most appropriate combination of a fluidizing and a stabilizing agent and optimizing the respective dosage
- a careful control of water quality and chemistry of the solution
- a deeper insight into the comminution process (size distribution and operational procedure and conditions)
- the assessment of the optimum degree of coal cleaning (ash content) and the study of the kind of washing operations before the CWS preparation (presence of flotation reagents, dewatering, …..).

Moreover the results obtained can encourage a feasibility study concerning the construction of a CWS plant near the Carbosulcis mine from which the fuel can be transported via pipeline to the nearby power station (about 3 km away) where it can be burned after minor modifications to the boiler. As an alternative, the CWS can be used for feeding a coal gasification plant often proposed for the environmentally friendly utilization of the high-sulphur Sulcis coal (5-6%).

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