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REVIEW ARTICLE

# The effect of soapstock on the cement raw mix grindability

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## KEYWORDS

Soapstock;  
Cement raw mix;  
Grindability;  
XRF;  
XRD;  
DSC

**Abstract** The aim of this work is to investigate the possibility of using industrial wastes as new grinding aids in cement industry. Two samples of Soapstock from different oils were used (Sunflower Oil (SO), Corn Oil (CO)). For this purpose, one reference sample was produced without using any admixture and another one using reference grinding aids (Triethanolamine TEA).

The raw mixes were characterized via differential scanning calorimetry (DSC), FT-IR technique, chemical analysis by X-ray fluorescence (XRF), X-ray powder diffraction (XRD), and mineral composition by Bouge equation and determination of free calcium oxide (CaO<sub>f</sub>) for clinker. Grindability was determined according to PSD and residues on sieve 90 μm.

In all cases the addition of grinding aids resulted in improvement of grindability (fact that was attributed to the additive ability not only to reduce resistance to combination, but also to prevent agglomeration and powder coatings of ball and mill), and the clinker produced was not effective by this admixture.

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## 1. Introduction

The grinding of raw material of cement manufacturing has broad effect on the homogeneity of feed raw material into rotation clinker furnace. Due to the effect of particle size distribution of raw material on melting and chemical reaction of clinker mixture, the grinding procedure was widely studied (Pilevneli et al., 2004; Jankovic et al., 2004). The grinding aid additives were improved via different organic material (this organic material has no effect on clinker composition) (Teoreanu and Guslicov, 1999; Yilmaz et al., 1993).

### 1.1. General view

The increasing of the world's population day by day and the development of the human relations in many aspects following the globalization of the earth have led to the increase in motion which in turn has expanded the world's energy consumption. This increase in energy consumption of the consumed energy has caused the investigation of new energy sources and the invention of novel less power-consuming technologies. The building sector is an indicator of the civilization level. This also shows the importance of cement to the civilization progress. Growing population and development both increase the need for cement production (Pilevneli et al., 2004). Production costs and environmental concerns are emphasizing the need to use less energy and therefore the development of more energy efficient machines for grinding and classification (Jankovic et al., 2004).

### 1.2. Facts

Cement world production currently accounts approximately 1.6 billion ton/year and the grinding process consumes nearly 2% of the electricity produced in the whole world. Furthermore, approximately 60–70% of the total electrical energy used in a cement plant is utilized for the grinding of raw materials, coal and clinker (Katsioti et al., 2009). The electrical energy consumed in the cement making process is in order of 110 kWh/tonne and about 30% of which is used for the raw materials preparation and about 40% for the final cement production by cement clinker grinding. For all dry grinding applications, cement production is certainly most important. Cement production process typically involves (i) grinding lime-

stone (and other raw materials to achieve the right chemical composition) to about 90% passing 90  $\mu\text{m}$  screen in a dry circuit, (ii) making cement by the chemical reaction between the components of the ground mixture. This chemical reaction occurs at high temperature in a rotary kiln. (iii) Grinding the cement clinker nodules to 100% passing 90  $\mu\text{m}$  screen in a dry circuit. (Jankovic et al., 2004)

### 1.3. Problem

As a result, a small gain in grinding efficiency can have a large impact on the operating cost of a plant. The efficiency of most grinding units is very low, where the raw material particles can coat the grinding media, can seal the armour plating and can agglomerate and form small plates which absorb the impact. The action of the grinding media within a rotating mill not only crushes the existing raw material particles, but also sharply compresses them, a fact that leads to the formation of electrostatic surface charges of opposed polarity. The material particles then agglomerate as a result of the forces of attraction acting on them. Consequently, the material particle agglomeration reduces the efficiency of the mill. This phenomenon is characterized by an increase in energy consumption whilst maintaining constant Blaine. The extent of agglomeration depends on many factors such as: (i) the specific characteristics of the materials to be ground, (ii) the operating parameters of the mill, (iii) the efficiency and distribution of the grinding media, (iv) the fineness of the raw material particles, (v) the internal operating conditions of the mill (humidity, temperature, ventilation, condition of the armour plating, etc.).

### 1.4. Solve

The agglomeration phenomenon remains one of the priorities of cement manufacturers, hence the importance of grinding aids (GA). The latter enables the partial neutralization of surface charges which have developed during milling. Additives, such as water, organic liquids and some inorganic electrolytes have been used to reduce the surface free energy of the material being ground with a view to improving grinding efficiency (Sohoni et al., 1991). Although the prime use of grinding aids are to reduce agglomeration of material particles, their use will also assist in: (i) the total or partial elimination of the "coating" effect on the media, (ii) an improvement in the separator

efficiency due to increased fluidity of fine particles, (iii) a decrease in pack-set problems in storage silos and bulk delivery trucks, (iv) an increased bulk and bag material quality, (v) improved materials-handling (blowing into silos, off-loading trucks etc) due to an improved fluidity, (vi) improved grinding production capacity. (Katsioti et al., 2009) (vii) significant mill output increase at the same fineness. The increase in production can be used to reduce production costs or to cover market demand. (viii) Fineness increase at equal output, or both effects. In some cases very high fineness may only be obtained by using grinding aids. (ix) Improved particle size distribution at equal fineness (Padovani, 2008). It is well known that the particle size fraction between 3 and 30  $\mu\text{m}$  is directly related to the burnability development of raw mix (Duda, 1985).

In the grinding process, a variety of grinding aids have been used. There are aliphatic amines such as triethylenetetramine (TETA), tetraethylenepentamine (TEPA) and aminealcohols such as diethanolamine (DEA), triethanolamine (TEA) and triisopropanolamine (TIPA). Glycol compounds are represented such as ethyleneglycol (EG), diethyleneglycol (DEG) Teoreanu and Guslicov, 1999. In addition, there are more complex compounds such as aminoethylethanolamine (AEEA) and hydroxyethyl diethylenetriamine (HEDETA). Phenol and phenol-derivates are also used as grinding aids, whereas, other compounds, such as amine acetate. Generally, the concentration range of grinding aids added is from 50 to 500 ppm. After the grinding process the additives might not be any longer in their original chemical form. In addition, grinding aid composition might not consist of mixtures of pure compounds, but rather more complex raw materials (Jeknavorian et al., 1998). Triethanolamine (TEA), a low tertiary alkanolamines, is used for various reasons in cement industry. The action of TEA in the TEA is a weak base and in an aqueous phase it is mostly in the molecular state. TEA has the ability to chelate with certain metallic ions such as  $\text{Fe}^{3+}$  in highly alkaline media (Katsioti et al., 2009; Yilmaz et al., 1993). The information in literature (Romiliat, 2006) indicates that the principal roles played by the GA are gets adsorbed on the surface of the material to be comminuted, i.e., either on the exterior surfaces or on the microcrack walls where it manages to enter. An immediate effect is a decrease in hardness accompanied by phenomena of adhesion and clogging. The reason behind the grinding aids's decreasing the hardness is based on Griffith's theory regarding fragile breakage, which postulates that fragile materials contain small cracks whose propagation may lead to material breakage. The Rebinder effect concept that a compound capable of selective adsorption inside the solid particle cracks undergoing grinding would impede the attraction between any residual electric forces, and in so doing it may impede any crack closure during the inactive phase. Taking the Rebinder effect to the next level, Tanaka maintains that the role played by the grinding aid on the ground material is similar to a wedge. The GA gets adsorbed on the surface of the material undergoing grinding and reduces the surface energy,

which only leads to further breakage. Moreover, the grinding aid hampers the adherence of the fine particles on the grinding media. All concepts suggested for the mechanism of the grinding aids depend mainly on the material undergoing grinding since they get adsorbed on the respective material. The adsorption on the inner surface layers of the ground material during the first phase of the process or the active phase presents a relative exception to the Traube–Duclaux rule. The adsorption on the inner surface layers may be significantly greater with the higher porosity of the material and indirect relation to the molar volume. The grinding aid it should also feature good lubricating properties. The lubricating properties are known to depend especially on the size of the hydrocarbon chain in the surfactant molecule (Teoreanu and Moanță, 2009).

## 2. Experimental

### 2.1. Materials

The investigations were carried out with limestone, basalt, sand were given by a local cement factory (Alarabia company, south Aleppo, Syria) which chosen as raw materials to prepare cement raw mixture, after a preliminary crushing stage carried out within the plant, the maximum size of limestone and basalt particles was smaller than 1 cm, respectively. Table 1 presents chemical composition of the raw materials, which were determined by XRF analysis (Table 1), then this materials were proportioned to prepared raw meal (26 kg) with (Lime Saturated Factor LSF = 98%, Silica Modulus SM = 2.31%) according to special program (the value commonly employed in the cement factory for the kiln feed), (Table 2 present raw meal percent).

Soapstock (SO): by-product produced from refining sunflower oil.

Soapstock (CO): by-product produced from refining corn oil.

(Coming from Sahteen & Afeia Company), and (Table 3) presents chemical composition of soapstock.

Triethanolamine (TEA) with Lab grade as reference material (purity 99%).

### 2.2. Experimental procedure and techniques

FT-IR measurement was done to Soapstock to identify organic material. We divided the raw meal into 13 samples each one 2 kg. grinding aids (GA) was introduced in variety percent (0.25%, 0.50%, 1.00%, 1.50%), then grinding was carried out in laboratory ball mill (capacity 5 kg) and the grindability was monitored by determining the percent of residue on sieve 90  $\mu\text{m}$  in internal time (30–60–90–120 min.) (where in cement plant raw meal residue in screen 0.090 mm is between 12% and 16%)

Raw meal particle size distribution (PSD) was determined as the % retained by sieving a sample of 10 g cement ground

**Table 1** The chemical composition of raw material.

Sample	L.O.I	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>	Cl	T.CO <sub>3</sub>	Moisture
Limestone	43.55	1.26	0.17	0.20	54.46	0.34	0.23	0.19	0.09	0.01	97.75	4.50
Basalt	14.16	42.81	11.28	9.59	11.80	6.41	2.35	0.74	0.07	0.03	15.03	5.62
Sand	1.86	94.96	0.44	0.43	1.50	0.20	0.19	0.11	0.19	0.01	2.27	2.17

**Table 2** Raw meal percent.

Sample	Result %
Limestone	74.10
Basalt	23.75
Sand	2.15

**Table 3** Chemical composition of soapstock.

Sample	Moisture %	Ash %	Unsaturated fatty acid %	Free acidity %	Acidic number
SO	38.64	8.66	50–52	0.08	0.17
CO	41.60	9.01	46–48	0.37	0.73

on 0.212, 0.090, 0.063 and 0.045 mm screen opening according to ASTM C430-96

In addition, the sintering reactions in all samples were recorded by means of differential thermal analysis using a Linseis DSC 851 instrument. The temperature was raised at a constant rate (10 °C/min) from ambient to 1450 °C. Mass sample was approximately 72 mg. The experiments were conducted in a static atmosphere.

The sintered samples were examined using a Siemens D-5000 X-ray diffractometer, with nickel-filtered Cu K $\alpha$ 1 radiation ( $\lambda = 1.5405 \text{ \AA}$ ) in order to identify the compounds formed during sintering.

The Bogue calculation was used to determine the mineralogical composition of the clinker produced as follows:

$$C_3S = 4.0710CaO - 7.6024SiO_2 - 6.7187Al_2O_3 - 1.4297Fe_2O_3$$

$$C_2S = 2.8675SiO_2 - 0.7544C_3S$$

$$C_3A = 2.6504Al_2O_3 - 1.6920Fe_2O_3$$

$$C_4AF = 3.0432Fe_2O_3$$

Free lime percent was determined using ACMEL device which contain electrode to measure the conductivity of mix (1 g of sample + 50 ml ethylene glycol).

### 3. Results and discussion

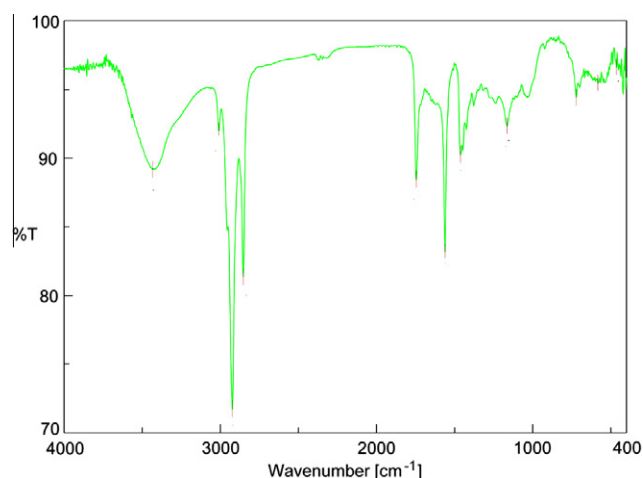
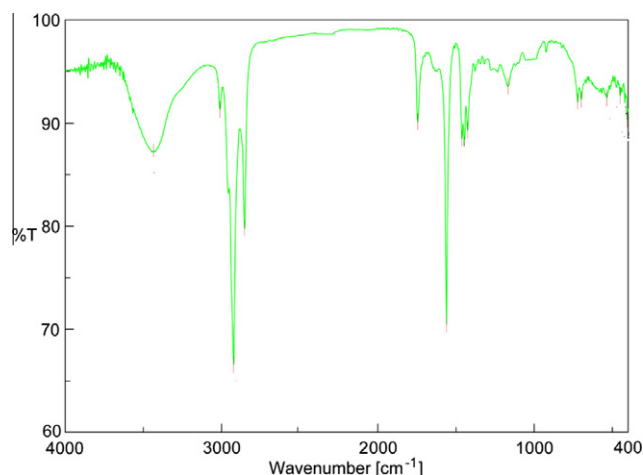
#### 3.1. Grinding aids characterization

Figs. 1 and 2 present the FT-IR spectra of the examined grinding additives. IR spectroscopy is generally used in order to give information as far as the composition of a sample and its structure. The bands occurring in the FTIR spectra of the examined additives can be characterized as follows:

The distinct broad band at 3600–3200  $\text{cm}^{-1}$  attributes O–H. The O–H stretching vibration is normally observed at about 3435  $\text{cm}^{-1}$ . The O–H in-plane bending vibration is observed in the region 1446–1260  $\text{cm}^{-1}$ .

The band at 3010  $\text{cm}^{-1}$  is due to the presence of =C–H stretching vibration in phenylic structures [UniqueID].

The band at 2925  $\text{cm}^{-1}$  is due to the presence of C–H stretching vibration in aliphatic structures, whereas the cor-

**Figure 1** FT-IR spectra of the examined grinding aids (Corn oil).**Figure 2** FT-IR spectra of the examined grinding aids (Sunflower oil).

responding at 2853  $\text{cm}^{-1}$  is attributed to the symmetrical CH stretching vibrations in  $-\text{CH}_2$ .

The band at 1750  $\text{cm}^{-1}$  is due to the presence of C=O stretching vibration in esteric structures. The band ranging from 1450 to 1500  $\text{cm}^{-1}$  stands for C–H<sub>3</sub> and C–H<sub>2</sub> distortion in all samples. The aliphatic C–H out-of-plane bending at 810  $\text{cm}^{-1}$  (together with the corresponding C–H stretching at 2853  $\text{cm}^{-1}$ ) suggested the presence of hydrocarbon species. In aromatic compounds, CH stretching frequencies appear in the range of 3000–3100  $\text{cm}^{-1}$ .

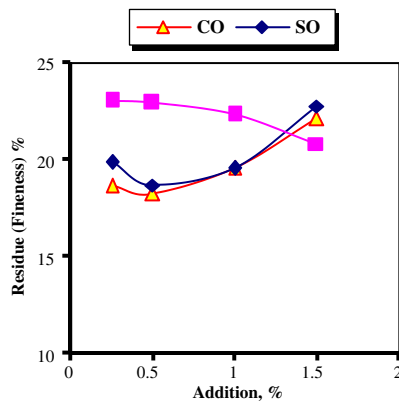
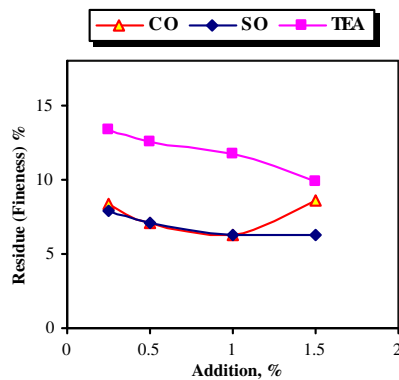
#### 3.2. Grindability study

Table 4 presents the evolution of the percent of residue on sieve 90  $\mu\text{m}$  of the raw meal at various grinding times in relation to the grinding aids type and ratio.

The investigations regarding the influence of the Soapstock (which is unsaturated fatty acid salts) in the process of grinding the raw meal have clearly highlighted the role of the surfactants in grinding. The best behavior highlighted by an decrease

**Table 4** Percent of residue on sieve 90  $\mu\text{m}$  of the raw meal (%).

Grinding time	Residue on sieve 90 $\mu\text{m}$ %												
	Control sample	Grinding aids ratio %											
		TEA				CO				SO			
		0.25	0.50	1.00	1.50	0.25	0.50	1.00	1.50	0.25	0.50	1.00	1.50
0	55.87	53.50	55.40	53.62	54.56	54.00	54.76	53.81	52.46	54.50	51.95	53.81	52.07
30	32.34	33.24	32.76	34.35	31.40	30.25	30.76	32.25	33.77	30.62	31.45	32.25	35.75
60	22.06	23.01	22.90	22.29	20.80	18.59	18.21	19.53	22.05	19.86	18.65	19.53	22.73
90	16.18	17.12	16.70	15.16	13.05	12.20	11.86	11.72	14.50	11.57	11.06	11.72	12.01
120	13.32	13.33	12.55	11.74	9.90	8.36	7.06	6.28	8.59	7.86	7.07	6.28	6.30

**Figure 3** The fraction retained on 0.090 mm screen opening after 90 min.**Figure 4** The fraction retained on 0.090 mm screen opening after 120 min.

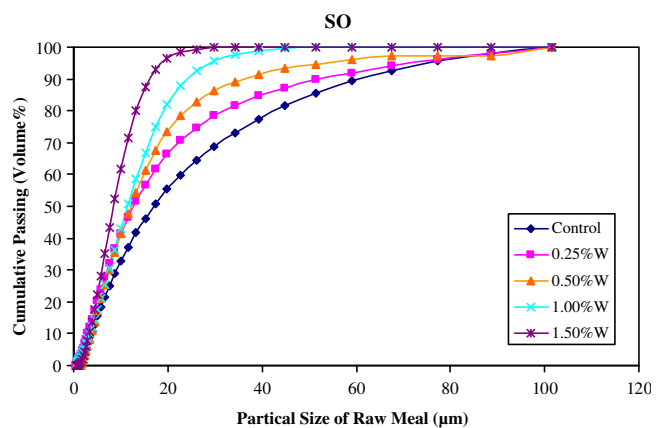
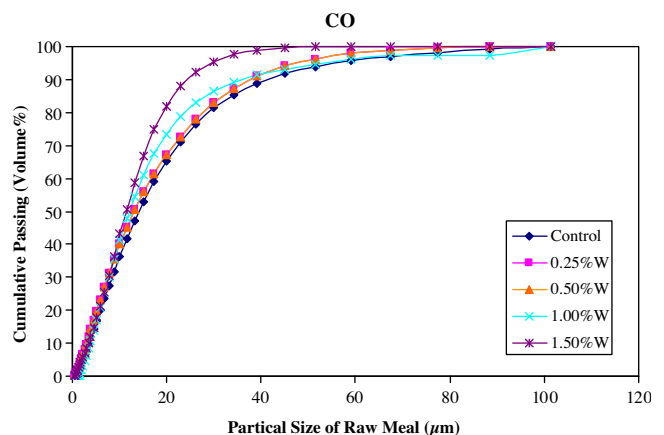
in the percent of residue on sieve 90  $\mu\text{m}$  at short grinding times of 60 min at the most was exhibited by a proportion of 0.5% CO which is 17% (Fig. 3). At grinding times longer than 120 min, residue was lesser in the case of employing SO, particularly in a ratio of 0.5% which is 32% (Fig. 4).

The justification of the above behavior may be viewed as a correlation between the phenomenon of adsorption of the Soapstock, the molecular weight of the Soapstock, and the condition of the granular material and narrower particle size range is generated as compared with raw meal without grinding agents. CO gets adsorbed far easier onto the grain pores at

short grinding times with its low molecular volume when compared to SO. Then again, at longer grinding times the Soapstock will rather get adsorbed on the grain surface layers. We gain better adsorption with Finer raw meal, the. An increase in Soapstock proportion influences the raw meal behavior in grinding. The best results were obtained with 0.5% SO.

### 3.3. Raw meal particle size distribution (PSD)

Fig. 5 and 6 present the evolution of the PSD diagram of the raw meal at various grinding aids type ratio. We notice the PSD diagram shift toward finer diameter.

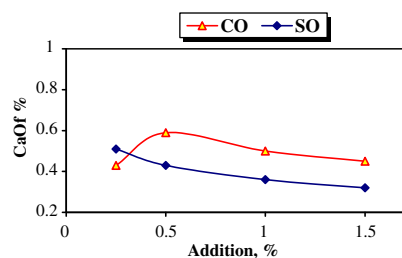
**Figure 5** The PSD diagram of SO.**Figure 6** The PSD diagram of CO.

**Table 5** Chemical composition of raw meal.

	Control sample	SO				CO			
		0.25%	0.50%	1.00%	1.50%	0.25%	0.50%	1.00%	1.50%
PC	35.63	35.38	35.40	35.27	35.44	35.38	35.85	35.4	35.65
SiO <sub>2</sub>	13.94	14.02	14.00	13.80	13.90	13.95	13.90	13.95	14.31
Al <sub>2</sub> O <sub>3</sub>	3.05	3.09	3.08	3.13	3.10	3.09	3.05	3.13	3.12
Fe <sub>2</sub> O <sub>3</sub>	2.65	2.72	2.76	2.81	2.80	2.72	2.66	2.83	2.78
CaO	43.50	43.71	43.67	43.45	43.70	43.85	43.50	43.80	44.80
MgO	2.67	2.73	2.65	2.71	2.66	2.68	2.64	2.70	2.68
Na <sub>2</sub> O	0.48	0.52	0.53	0.57	0.50	0.52	0.52	0.55	0.57
K <sub>2</sub> O	0.15	0.15	0.15	0.16	0.15	0.15	0.15	0.16	0.16
SO <sub>3</sub>	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Cl	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
A.R	1.15	1.14	1.12	1.11	1.11	1.14	1.15	1.11	1.12
H.M	2.21	2.20	2.20	2.20	2.21	2.22	2.22	2.20	2.22
L.S.F	98.32	98.09	98.09	98.64	98.36	98.84	98.56	98.46	98.58
S.R	2.45	2.41	2.40	2.32	2.35	2.40	2.43	2.34	2.43

**Table 6** The CaOf content of the prepared samples after thermal treatment.

Sample	%	The percent of residue on sieve 90 $\mu$ m	Blain (cm <sup>2</sup> /gr)	CaOf % 1400 °C 20 min
Control	—	6.30	3221.76	0.79
SO	0.25	5.60	3429.64	0.51
	0.5	4.40	3656.73	0.43
	1	4.00	3879.05	0.36
	1.5	3.90	3990.10	0.35
CO	0.25	5.60	3302.10	0.43
	0.50	6.00	3448.01	0.59
	1.0	6.30	3638.38	0.50
	1.5	6.00	3909.52	0.45

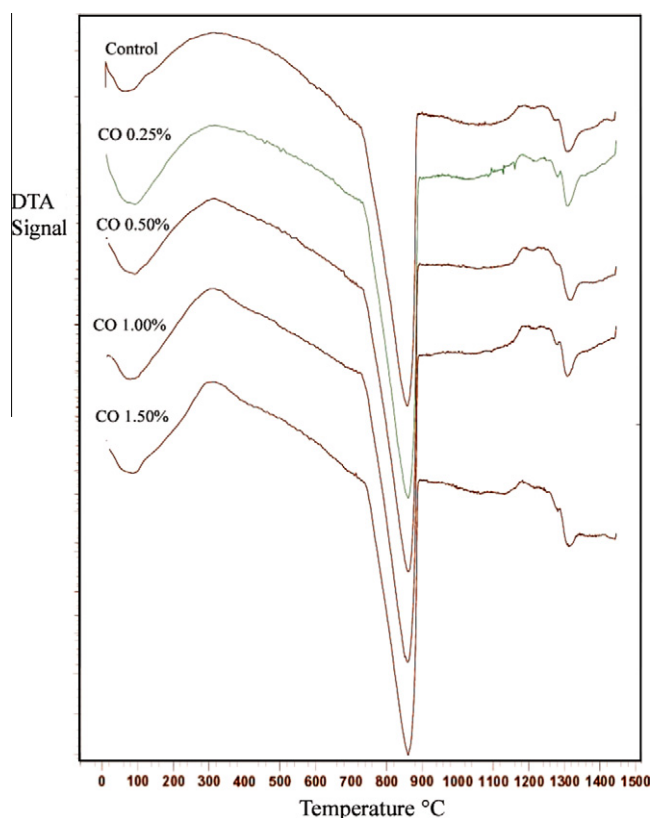
**Figure 7** The CaOf content in relation to the kind and percent of soapstock.

In the case of SO with increase the percent of addition the finer diameter increase until we reach ratio 98% of particle have diameter less than 20  $\mu$ m in the addition of (1.5 wt%), whereas in control sample 95% from particle size less than 80  $\mu$ m, because of high reactivity which have these material (Soapstock) in dispersion of raw material that agglomerate together then subject to further grinding whereas raw material which have no grinding aids agglomerate and trap the coarser material. The addition of CO have less effect than SO, we find that 98% from particle size less than 38  $\mu$ m because of SO have fatty acid unsaturated more than CO.

### 3.4. characterization and Burnability of the raw mix

The characterization of raw meal was done to all samples by chemical analysis by XRF. Table 5 shows the chemical composition.

The reactivity of the raw mixtures was evaluated on the basis of the unreacted lime (CaOf) content after sintering at fix temperature after grinding all sample to constant residue and Blain. Table 6 shows the CaOf content of the samples after the thermal treatment. Fig. 7 presents the CaOf content in relation to percent of Soapstock.

**Figure 8** DSC pattern to corn soapstock.



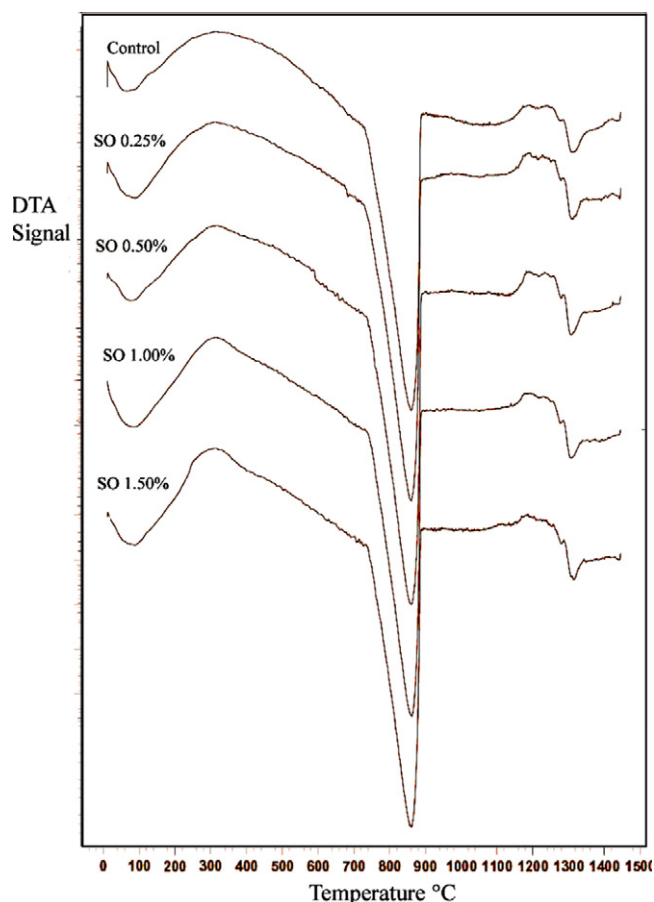


Figure 9 DSC pattern to sunflower soapstock.

We notice CaOf content in relation to the kind and percent of Soapstock decrease which improve the burnability of raw meal. Because of the soapstock contain some ash (Table 3), which acts as minerals in burning of raw meal so increase the combination of CaO with other components of raw meal, and reduce free CaO.

### 3.5. DSC studies of the raw meal

The thermal characterization of raw meal was done to all samples by DSC. Figs. 8 and 9 show the pattern of DSC to corn and sunflower Soapstock. The decomposition temperature of  $\text{CaCO}_3$  for all samples was the same, also the same temperature to formation of silicate phase and liquid phase, as it is shown in Table 7, so the Soapstock from two sources do not affect on clinkering reaction.

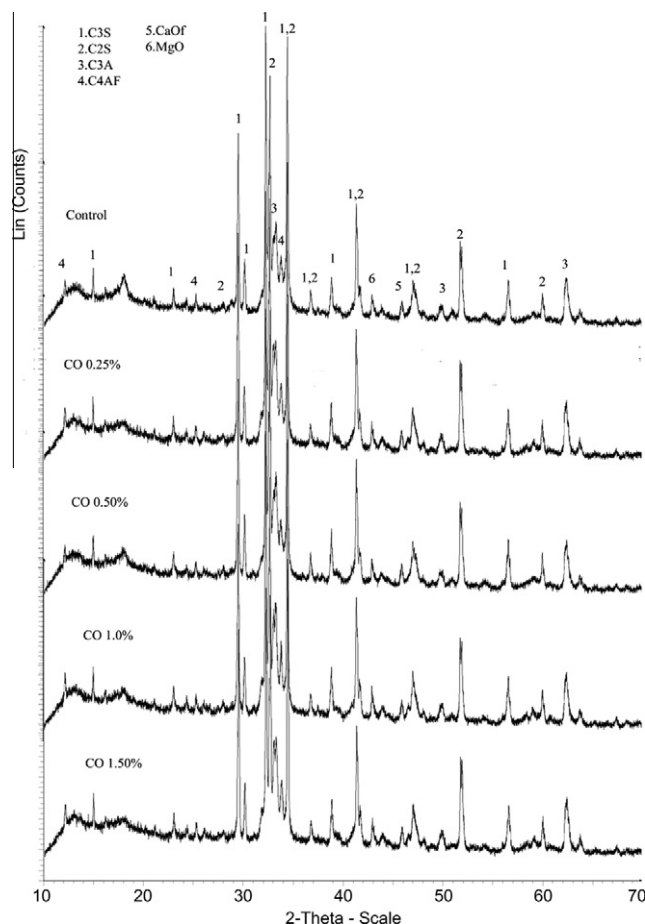


Figure 10 X-ray diffraction of corn soapstock.

### 3.6. XRD studies and Bogue calculation of the clinker

The XRD analyses of the produced Portland clinkers are shown in Figs. 10 and 11. As can be seen, the addition of the Soapstock did not affect the formation mineralogical phases of the produced clinker. In all clinker types, the dominant phases (alite, belite, calcium aluminate and ferrite) were well-crystallized giving peaks at the expected  $2\theta$  values. Additionally, the XRD patterns of the samples did not show any indications of formation of new phases, probably because of the extensive overlapping of the peaks and/or the low doping concentration. Finally, the alite phase in both cases has the monoclinic form.

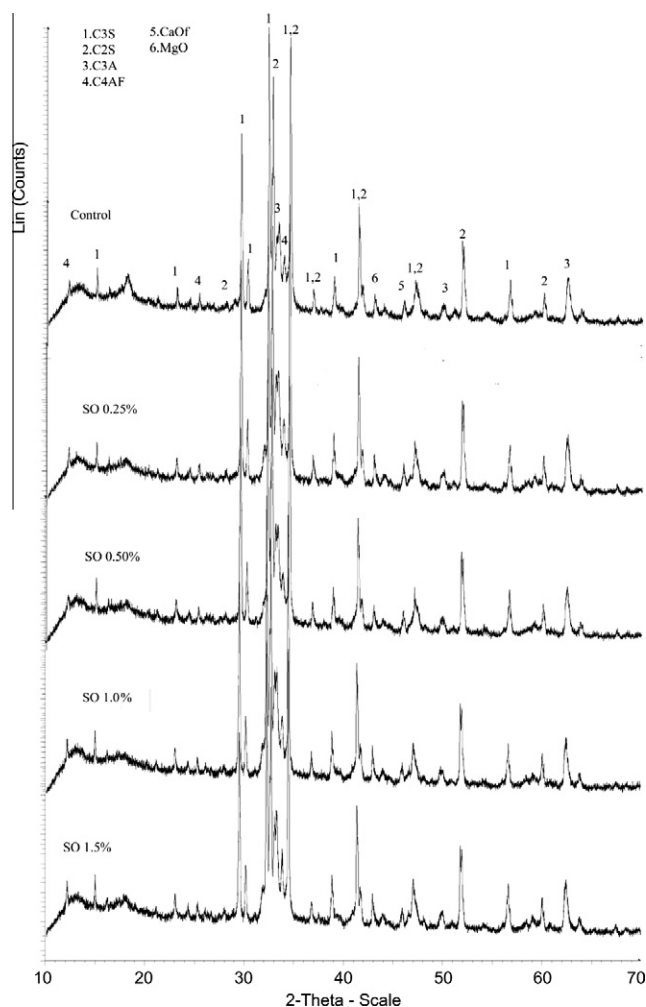
Table 8 shows the Bogue calculation of the clinker. We notice that all material does not affect the clinker composition.

Table 7 The DSC characterization of the prepared samples after thermal treatment.

Kind of reaction	Control sample	Grinding aids ratio %							
		CO				SO			
		0.25	0.50	1.00	1.50	0.25	0.50	1.00	1.50
Moisture	89.1	91.3	91.6	81.2	83.7	89.3	81.4	80.5	81.3
Organic material	—	242	293.6	294.5	288.4	—	291.1	295.2	293.5
$\text{CaCO}_3$ decomposition	860.1	860.7	861.6	860	860.9	861.2	860.4	860.5	861.3
Silicate phase	1188.6	1182.8	1189.1	1199	1181.8	1190.2	1189.2	1188.3	1186.5
Liquid phase	1314.9	1310.3	1317.6	1310.3	1312.7	1313.7	1307.9	1306.5	1310.8

**Table 8** The Bogue calculation of the clinker.

	Control	CO				SO			
		0.25%	0.50%	1.0%	1.5%	0.25%	0.50%	1.0%	1.5%
C <sub>3</sub> S	70.28	71.19	70.73	70.09	70.91	71.19	70.73	70.09	70.91
C <sub>2</sub> S	7.11	6.00	6.61	6.69	6.45	6.00	6.61	6.69	6.45
C <sub>3</sub> A	5.41	5.35	5.39	5.22	5.20	5.35	5.39	5.22	5.20
C <sub>4</sub> AF	12.11	12.34	12.16	12.81	12.34	12.34	12.16	12.81	12.34

**Figure 11** X-ray diffraction of sunflower soapstock.

#### 4. Conclusion

From the present study the following conclusion can be drawn:

1. The raw meal particles with SO and CO are finer, while those without GA are coarser. Accordingly, GA prevents agglomerates the raw meal grains.

2. The PSD of the raw meal narrows when the addition of SO and CO into raw meal is increased. That's, as the addition of SO is increased, the granulometric range widens.
3. SO and CO don't affect on Burnability of clinker and mineralogical phases of the produced clinker. Inversion of that its improve Burnability of clinker. Because the GA including some salts.

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