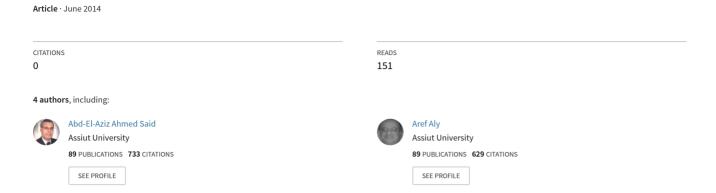
# Utilization of lime mud as a filler in paper making



#### Assiut University Journal of Chemistry(AUJC) 43(2014) 41-46

Journal homepage: www.aujc.org

ISSN 1678-4919

Vol(43) No(1-2) 2014

# Full Paper

# Utilization of lime-mud as a filler in paper making

Abd EL Aziz A. Said  $^{(1)}$ , Aref A. M. Aly  $^{(1)}$ , Adel Abd EL Hameed Ahmed  $^{(2)}$ , and Hazem S. Ahmed  $^{(2)}$ 

(1) Chemistry Department, Faculty of Science, Assuit University, Assuit Egypt

Email: aasaid55@yahoo.com

Article history: Received: 26/9/2014; Revised: 23/10/2014; Accepted: 27/10/2014

; Available Online: 11/11/2014

**Abstract:** The utilization of lime-mud as a filler in paper making was studied. The lime – mud before and after treatment were characterized using laser scattering particle size distribution technique. The dregs and alkali metal losses were removed. The final samples were grinded to achieve the proper particle size. Finally, the treated lime-mud was applied in the research plant as filler on paper sheets.

**Keywords:** Lime-mud, particle size, filler, paper making.

#### 1. INTRODUCTION

Pulp and paper industry is a source of a wide range of solid inorganic and organic wastes generated during the various processes in bleached Kraft production. Waste handling receives a great concern in pulp and paper industrial plants either by minimizing the generation of this waste or to reuse it, whenever practicable.

Bagasse, the most fibrous raw material available and suitable for paper making in Egypt, is the sugarcane residue after sugar extraction. The ultimate goal of chemical pulping is to liberate cellulosic fibers by lignin removal (delignification) without degrading or removing fibers. Alkaline pulping processes are not totally selective for lignin, and cellulose is degraded by the cooking liquor [1].

In the Kraft pulping process, bagasse mixed with aqueous cooking liquor white

liquor" composed mainly of sodium hydroxide (NaOH) and sodium sulfide (Na<sub>2</sub>S). This mixing occurs in a "digester" vessel at a temperature and pressure satisfactory to separate the cellulosic fiber from the natural lignins that bind such fibers. The dead load chemicals have no direct effect on pulping. An increase in dead load can result in a decrease in lignin removal in pulping [2].

The liberated fiber is separated from the resultant "black liquor" and is subsequently washed, screened and bleached and eventually transformed into paper. Washing filters remove the maximum amount of diisolved organic and soluble inorganic material present with pulp mass to prevent any interference with bleaching or papermaking chemicals [3].

Bleaching is the chemical treatment of cellulosic fibers to increase brightness by

<sup>(2)</sup>Quena Paper Industry Company

decolorizing or dissolving the colored components in pulp, principally lignin to increase the visual quality [4]. Bleached pulp mixed in a furnish with wood pulp and other chemical additives (filler, dye, retention aid, AKD, starch) to produce the paper roll [5].

The separated black liquor contains, aside from the original white liquor chemicals, lignins and other organic matter. In order to recover and recycle these costly pulping chemicals, as well as to produce valuable pulp mill steam and power from the contained organic lignins, the black liquor is concentrated in multiple-effect evaporators and delivered as a concentrated fuel to a "chemical recovery boiler."

This recovery boiler combusts the organics under unique oxidizing/reducing conditions to a molten inorganic ash ("smelt") consisting primarily of Na<sub>2</sub>S and Na<sub>2</sub>CO<sub>3</sub>. The coproduced high-pressure steam is subsequently exhausted via a steam turbine/generator to produce mill power and lower pressure mill process steams [6]. The smelt is drained from the recovery boiler and quenched in water to create the "green liquor." This green liquor is subsequently clarified and filtered to remove the insoluble impurities whereupon it is delivered to the "slakers" to initiate conversion of the dissolved Na<sub>2</sub>CO<sub>3</sub> into NaOH required in the white liquor. This slaking process utilizes calcium oxide CaO to convert Na<sub>2</sub>CO<sub>3</sub> into NaOH via the following two consecutive reactions:

$$CaO(s) +H_2O \rightarrow Ca (OH)_2 \qquad (1)$$
 
$$Na_2CO_3 (aq) + Ca(OH)_2 \rightarrow 2NaOH(aq) + CaCO_3(s) \quad (2)$$

CaCO<sub>3</sub> is the lime mud waste produced from chemicals recovery in pulp and paper manufacture and can be used as filler of paper [7-9]. There are various studies on lime mud waste recycle, among which are land application of lime mud [10] and lime mud combustion to produce CaO [11-12]. Therefore, the aim of the present study was to make use of the lime mud waste with a particle size suitable as filler in paper making.

#### 2. Experimental

Lime-mud obtained from the recovery plant was dried and then the alkali loss was measured. It was subjected to full chemical analysis, particle size determination and optical properties. The study included the effect of washing with hot water at different temperatures on the removal of alkali and dissolved silica then full chemical analysis was conducted. The effect of grinding on the particle size distribution using constant volume and changing the time of grinding was also investigated. The optical properties brightness, whiteness, which include: yellowness, opacity, scattering coefficient were determined as well.

Lime-mud, calcium carbonate and calcium oxide raw were analyzed according to Tappi Standard Methods for pigments sampling  $T_{657}$  om- $_{92}$ , free moisture in filler and pigments  $T_{671}$  cm- $_{85}$ , analysis of lime  $T_{617}$  cm- $_{84}$ , analysis of calcium carbonate  $T_{660}$  wd- $_{79}$ , brightness of pigments  $T_{646}$  om- $_{86}$ , opacity  $T_{425}$ , and particle size distribution of coating pigments  $T_{649}$  cm- $_{90}$ .

#### 2.1. Materials

Lime-mud, calcium carbonate and calcium oxide raw materials samples are used in this study.

#### 2.2. Apparatus

- Flame photometer, JENNWAY, for measuring Ca, K, and Na.
- HORIBA laser scattering particle size distribution analyzer LA-300.
- HORIBA LA-300 grinding mill
- Thermolyne TZ4ST furnance.
- Labtech instruments inc. standard sheet machine, automatic sheet press 400-1
- Technidyne laboratory services (TLS) model ISO for measuring optical properties.
- Roughness tester (BENDTSEN method) for measuring air permance.
- Burst PTI laboratory, equipment Laborausrustung, range 50 bars.

- ELMENDORF tearing tester, THWING ALBERT instrument.
- Universal tensile tester, KARL SCHROODER Kg. D-69450.

All measurements were performed on the above mentioned instrument and apparatus available at the Qena Paper Industry Company

#### 3. Results and Discussion

#### 3.1.On the laboratory scale

# a. Characterization of lime-mud before and after treatment

**Table 1** shows the results of the chemical analysis of the lime mud (waste generated in Quena Paper Industry Company) after drying at 100°C, and the other samples after washing under continuous stirring. Lime-mud to water volume used in washing is 1:2.5 and after the disposal of water washing and drying the sample was analyzed again as shown in Table 1. The results illustrate the significant reduction in the percentage of soluble silica and the increase in CaO%.

## b. Washing of lime-mud to remove alkali

**Table 2** shows that the chemical loss of Na<sub>2</sub>O decreases from 1.45% to 0.55% which then becomes more suitable for paper making. From Tables 1 & 2, it is clear that washing leads to lowering of the alkali and soluble silica.

**Table 2:** lime-mud samples before filter, lab washing and chemical loss measuring.

Temperature	60°C	70°C	80°C	90°C
Loss as Na <sub>2</sub> O%	1.45%	0.86%	0.84%	0.55%

## c. Grinding

**Table 3** shows a small increase in  $2\mu m$  portion with grinding of lime-mud samples without treatment, and high increase in  $2\mu m$  portion with grinding lime- mud samples after washing, drying and grinding in order to soften the particles size, in comparison with CaCO<sub>3</sub> filler sample. These facts are depicted in Fig. 1.

**Table 1:** Chemical analysis for samples before and after treatment (%)

sample	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>	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	L.O.I
Dried sample	4.85	0.01	0.03	51.13	0.01	1.45	0.01	0.39	0.01	0.11	40.95
Washed, dried sample	3.50	0.01	0.02	54.39	0.01	0.55	0.01	0.01	0.01	0.10	41.28

**Table 3:** Particle size distribution before and after treatment at different grinding times (CaCO3 \* is the currently fillerused in the factory)

	Raw sample direct from the filter	Drying and grinding for1 min	Washed, drying and grinding for 5 min	Washed, drying and grinding for10 min	CaCO <sub>3</sub> * Filler sample
Median μm	15.1	4.3	0.6	0.5	1.6
Diameter on 10% μm	0.3985	0.3876	0.2179	0.2	0.7
Diameter on 50% µm	15.1	4.3	0.6	0.5	1.6
Diameter on 90% μm	29.4	10.3	9.3	6.3	3.7
% On diameter 2µm	15.0 %	32.7 %	70.5 %	71.7 %	73.2%
% On diameter 3µm	15.7 %	39.7 %	74.3 %	77.8 %	95.1%
% On diameter 4µm	16.7 %	47.5 %	77.4 %	82.4 %	99.4%
% On diameter 5µm	17.9 %	55.9 %	80.3 %	86.4 %	
% On diameter 10μm			90.9	95.903	
Mean μm	15.7	5.3	2.8	2.0	1.9
Brightness iso		90.2		92.7	97.8%
d98 (Top cut)			18.2	12.4	6.9

# 3.2. On the pilot scale

### Paper making research plant

From the above results it can be concluded that the lime mud samples after treatment and grinding become suitable to be used as filler in paper making.

In the following (Table 4) are the properties of paper sheets using treated lime mud as a filler together with those of commercial calcium carbonate as a filler.

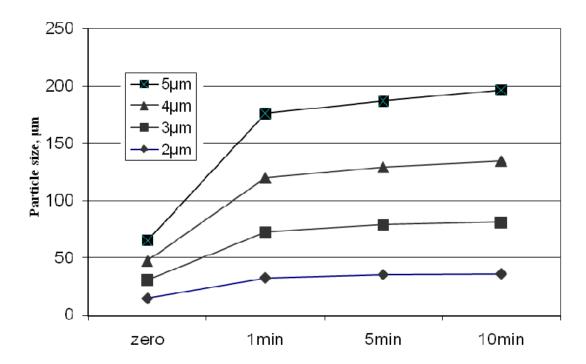


Fig.1. Relation between grinding time and particle size

**Table 4:** Optical and mechanical properties for paper sheets, where sample1 is the paper sheets made using the commercial calcium carbonate as a filler and sample 2 represents the paper sheets made by using treated lime mud.

		Sample 1	Sample 2
Substance	g/m2	66.03	62.94
Thickness	μ	400	390
Bulk	cc/g	1.51	1.55
Moisture	%	6.67	5.88
Tensile index	Nm/g	53.97	58.64
Breaking length	METER	5500	5980
Tear index	mNm2/g	7.27	7.01
Tear factor		73.93	71.52
Burst index	Kpa m2/g	3.93	3.98
Burst factor		40.06	40.56
Air permeance	ml/min	227	206
Opacity (ptg)	%	80.89	78.73
Sc Coefficient	m2/kg	36.99	34.15
Yellowness	%	5.96	4.65

#### Conclusion

Tests made in this study showed that lime-mud after washing and drying is an important source of crude carbonate which (due its chemical properties and natural potential) can be used in paper making industry as a filler in coated paper with the following superior properties:

- Some chemical contents and dregs were removed.
- Brightness is suitable for papermaking.

Lime-mud particles size becomes smaller and percent of 2  $\mu$ m (15.0%) increases gradually with increasing time reaching 71.7 % and thus becomes the carbonate more suitable for paper making.

#### References

- 1. C. H. Matthews, Svensk Papperstid. 77(1974)629.
- 2. A. Teder, and L. Olm, Paperi puu 4(1981)315.
- 3. J. H. Perkins, H. S. Welsh, and J. H. Mappus, Tappi 37(1954)83.
- 4. D. W. Reeve, Introduction to pulp bleaching study guide, Home Study Library, TAPPI Press, Atlanta, 1986.
- 5. W. H. Griggs, and B. W. Crouse, Tappi 63(1980)49.
- 6. P. Gundersby, Proceeding, Intl. Conf. on the Recovery of Pulping Chemicals, TAPPI/CPPA, 1985.
- 7. J. Wang, P. Liu, W. Sun, Bioresour. 7 (2012)5894.
- 8. H.Konno, H. Goto, K. Takahashi, Y. Nanri, TAPPI PaperCon'09 Conference, St. Louis (2009)1.
- 9. J. Wang, D. Zhang, Y-J. Xu, Ippta 26 (2014)88.

10. J. K. Muse and C.C. Mitchell. Agronomy Journal 87(1995)432.

- 11. J. P. Gorgo, T. N. Adams, Design and performance of rotary lime kilns in the pulp and paper industry, Atlanta (1986).
- 12. G. M. Dorris, J. Pulp and Paper Sci. 29(2003)185.