Quality Test of Rice Hull Ash Filter Aids

Wenping Li, Carl Kiser, Quintin Richard Agrilectric Research Company P. O. Box 3716 Lake Charles, Louisiana 70602 Phone: 337-430-0006 Fax: 337-421-6344 Email: Wenping_li@frmco.com

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ABSTRACT

Rice hull ash (RHA) is produced from the combustion of rice hulls for energy generation and steam processes. This product is an excellent filter aid quantified by the very good physical, chemical as well as filtration properties, such as fine, porous, rigid, easily dispersed and suspended, and chemically inert. Quality of the filter aid is determined by its physical and chemical properties, and is reflected by its filtration performance, which is evaluated by cake permeability, filtrate clarity, solid rejection rate, wt% of cake solids in filter aid precoat filtration, and filtration of solid-liquid suspensions with filter aids as a body feed. Standard methods of testing filter aids quality include pH, particle size, bulk density, permeability, solid rejection rate, and moisture of cake. Test results of different grades of RHA filter aids are presented.

KEYWORDS

Rice Hull Ash (RHA), Suspension burned RHA, filter aids, filtration, permeability, rejection rate, filtration efficiency, quality test

INTRODUCTION

<u>Filter Aids</u>

Filter aids are used to improve filtration of colloids, extra fine particles, gel like, and highly compactible materials, which are difficult to be filtered or deliquored due to low filtrate rate, unacceptable filtrate clarity, high cake moisture content, or serious filter medium clogging. Proper dosage of filter aids as a "body feed" with difficult to filter slurries will increase cake porosity and permeability, decrease cake compactibility, and sometimes help to increase cake porosity but decrease average pore size so as to improve both filtrate clarity and filtrate rate.

Diatomaceous Earth (DE) filter aids have been widely used in food, beverage, chemical and mineral industries in the past 70 years. It is mined from deposits of small skeletal remains of aquatic plants from either fresh or salt water origins. Sources for diatomaceous earth filter aids are somewhat limited due to restrictions on type and shapes of DE deposits, and contamination by clay, sand, iron oxides, etc (Kruining, 1988). As an

alternative, Rice Hull Ash (RHA), a porous silica material (Figure 1) produced from renewable biomaterials is very similar to some forms of DE filter aid products, and possesses excellent physical, chemical and filtration properties.



Figure 1. Suspension Burned RHA Porous Structure

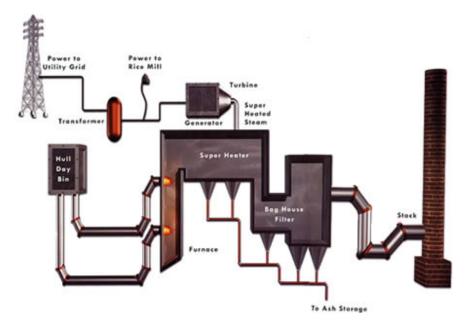
Rice Hull Ash Filter Aids

Rice Hull Ash (RHA) is produced from a renewable resource rice hulls, which in the past, was a disposal problem in the rice milling industry. Rice hulls naturally have high silica content (around 20%), high carbon content which enables them to be used as a fuel, and a porous structure (Houston, 1972), which produces a highly porous silica ash after the hulls are burned.

There is a wide range of RHA physical and chemical properties depending on different rice hull burning processes, soil conditions, and fertilizers used. The Suspension Burning Process (Agrilectric International Technology brochure) generally produces more amorphous silica and less crystalline silica than other combustion processes (Bronzeoak Ltd., 2003). This paper focuses on the RHA produced by the suspension burning process.

In the suspension burning process (Figure 2), processed raw rice hulls are pneumatically conveyed into the furnace as a fuel for electricity generation or process steam. Temperatures in the furnace range from 1000°F to 2500°F for various times ranging from 2 seconds to 5 minutes. Upon leaving the furnace, the combustibles are consumed and the ash is rapidly removed for further particle processing, such as grinding, screening, air classifications, or special surface treatments. Different grades of RHA products are produced for varying filtration applications.

RHA produced from the suspension burning and following particle processes are porous, high amorphous silica particles with appropriate size and shape, low in crystalline silica and carbon contents, and exhibit excellent filtration properties. For the purpose of this report, Suspension Burned RHA product will be used as the standard for this product. Physical, chemical, and filtration properties affecting quality of RHA filter aid



products, and corresponding test will be discussed.

Figure 2. Rice Hull Ash Suspension Burning Process

CHARACTERISTICS AFFECTING RHA FILTER AID QUALITY

Characteristics of RHA filter aids include chemical, physical and filtration properties as summarized in Figure 3. Permeability, cake moisture content, and filtrate quality are used to evaluate performance of filter aid products. Typical physical and chemical properties affecting filtration performance and product quality of a filter aid include chemical composition, crystalline silica content, bulk density, particle size distribution (PSD), particle shapes and particulate pattern, and porous structure.

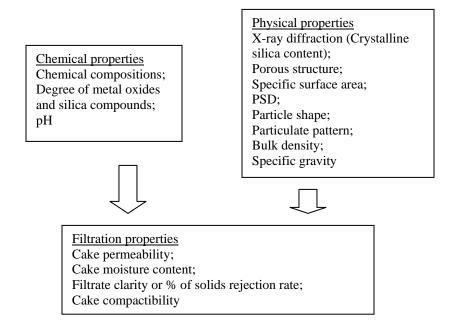


Figure 3. RHA Filter Aids Characterization

Chemical Composition

If properly controlled, suspension burned RHA filter aid products are composed of high amorphous silica and carbon concentrations around 2-8%. A typical chemical analysis is shown in Table 1.

Principal minor ingredients include aluminum, calcium, iron, potassium and magnesium. Normally, soluble metal oxides are considered sources of contaminants to filtrate product. Fortunately, most metal oxides in RHA are combined as silicates and not readily soluble.

Existence of fine carbon particles may cause turbid filtrate during initial cake formation stage. They may also perform as an adsorbent for small molecules impurities removal.

Ash Analysis	Range*	
Ash Ahaiysis Carbon Silicon Dioxide Other By Weight % Aluminum Oxide Ferric Oxide Calcium Oxide Magnesium Oxide Sulfur Trioxide Sodium Oxide Potassium Oxide Titanium Oxide Phosphorus Pentoxide	2.09—7.59 89.65—96.9 0.006—0.039 0.006—0.052 0.48—0.81 0.13—0.53 0.018—0.24 0.018—0.18 1.74—2.69 0.003—0.02 0.74—1.23	
Manganic Oxide	ND-0.20	

Table 1. Properly Controlled Suspension Burning RHA Chemical Composition

*Note: Results are based on 256 Independent laboratory samples over operating history of the Agrilectric power plantfrom 1984-2004.

Crystalline Silica Content

In the rice hull combustion process, crystalline silica is produced due to high temperature or long combustion time. Higher crystalline silica content of RHA is an indication of less porous structure. Furthermore, crystalline silica has been considered hazardous to human respiratory system. It is desirable to have low crystalline silica concentrations in all filter aid products including RHA. The properly burned RHA contains less than 1.5% crystalline silica determined by X-ray diffraction method.

Bulk Density

Compared to other physical or chemical properties, bulk density can be measured quickly and easily. With consistent particle specific gravity and ignoring particulate surface charge, bulk density provides an indication of particle size. The finer the particles, results in higher bulk density. Bulk density of processed hulls and suspension burned RHA ranges from $16-22b/ft^3$.

Particle Size

It is generally known that the finer the particles, the lower the permeability and the higher the filtrate quality. However, different results were observed on RHA filter aid based on recent research. Five samples with decreasing particle sizes are compared with their corresponding permeabilities and filtrate turbidities as shown in Table 2. From sample 1 - 4, cake permeability decreases when particle size is decreasing with no obvious improvements to filtrate clarity. Unexpectively, when particle size decreases to sample 5, there is a tremendous increase of cake permeability and decrease of filtrate turbidity. Explanations lie in different particle shapes and particulate pattern of sample 1 to 4 and sample 5. When comparing the effect of particle size on filtration performance, particles should be with the same shape and particulate pattern.

Sample #	Particle Size	Filter Cake Permeability*, darcy	Filtrate Turbidity*, NTU
1	П	0.17	32.8
2		0.16	24.1
3		0.11	29.4
4	ረታ	0.098	33.3
5	V	0.72	2.36

Table 2. Effect of RHA Particle Size on Permeability and Filtrate Clarity

• Constant pressure filtration at 15psi

Particle Shape and Particulate Pattern

SEM studies of RHA indicate existence of two types of particulate patterns -Pattern I as shown in Figure 4, and Pattern II as shown in Figure 5. Particles of Pattern I are mostly porous and mostly with regular shapes. Particles of Sample 1 to sample 4 fall into this pattern category. On the other hand, particles of Pattern II are very irregular in their shapes, and contain partially porous and partially non-porous particles. Irregular shaped particles tend to form a filter cake of higher porosity and cake permeability compared to regular shaped particles, which explains higher permeability of sample 5.

Due to porous structure of particles for samples 1 to 4, in a particulate cake structure, there are not only inter-particle pores, but also intra-particle pores. Intra-particle pores are not affected by particle size, and those pores may dominate pores of a filter bed as the regular particles are condensed. Therefore, filtrate turbidities from filtration of samples 1 to 4 are close.

Smaller particles of sample 5 with smaller inter-particle pore size compared to samples 1 to 4 of larger inter-particles and intra-particle pore size gave an explanation of better filtrate turbidity of sample 5.

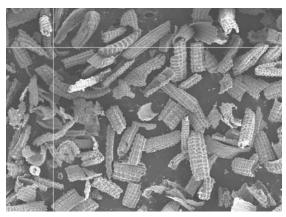


Figure 4. Pattern I of Rice Hull Ash Filter Aid

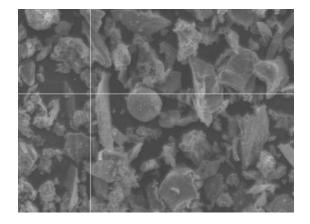


Figure 5. Pattern II of Rice Hull Ash Filter Aid

RICE HULL ASH FILTER AID QUALITY TEST AND RESULTS

Practically, it is difficult to characterize particle shape and porous structures, and it is not necessary to quantify chemical compositions for daily RHA quality monitoring and control. As has been discussed, quality of RHA filter aid products is affected by those chemical and physical properties but is reflected by permeability, filtrate clarity or % solid rejection, and moisture of cake after filtration. With a good representative sample, filtration tests along with pH, bulk density and PSD should provide sufficient information for product specification and quality monitoring.

<u>pH Test</u>

Soluble metal oxides may be a cause of filtrate product contamination, and has been a consideration for selection of different filter aids for some specific applications. pH as an indirect reflection of soluble metal oxides, is easy to be measured, and is included in the product quality test. 200grams 5 wt% of filter aid suspension mixed at 200rpm for 10 minutes are used in the test. Variation of pH may be a factor affecting filtration performance of RHA product.

Particle Size Distribution

Simple sieve analysis using various mesh screens provides valuable information on distributions and classifications of different size range particles. Values of d10, d50 and d90 based on cumulative particle size distribution curve are used to characterize particle size.

Bulk Density

Bulk density is determined by a modification to ASTM C-29.

Permeability, Filtrate Turbidity, and Moisture of Cake

A simplified permeability test method in a 0.0045m² filter cell under 15psi constant filtration pressure has been developed to measure cake permeability, cake moisture and filtrate turbidity (Li, Kiser and Richard, 2005). Suspensions containing filter aids only are used in the test. Permeability is a reflection of physical properties such as particle size, shape, particulate porous structures, and chemical properties. Permeability is normally an important filter aid product specification.

Turbidity of filtrate during filter aid permeability test is included as a specification in product quality test. It can be an indication of solid rejection efficiency of a precoat filter aid bed. It can also be an indication of the rate at which a filter cake is initially built on the surface of filter media. The faster the cake is built, the lower the filtrate turbidity.

Moisture of the filter aid cake is also obtained from the permeability test. It is included in the specification and quality test as an indication of dewatering properties of a filter aid product.

% Solids Rejection

Percentage of Solid rejection is defined by % of total solids rejected by a filter aid precoat. A standard test is designed to measure % solid rejection using 5 mm precoat (formed under 15psi) followed by filtration of 0.5% ISO 12103-1 A2 Fine Test Dust (Peuchot, 2000), with 0.5% RHA filter aid as a body feed. Total suspended solids in filtrate are measured and used to calculate the % of solid rejection by the 5mm precoat and 0.5% body feed.

Test Results of Three RHA Filter Aid Products

Typical test results on three grades of RHA filter aid products are shown in Table 3. Comparing d10, d50 and d90 data in Table 3, the particle size comparison is Grade III

> Grade I > Grade II. Correspondingly, bulk density of the three grades is Grade III < Grade I < Grade II.

Different grades of the three products contain different ratios of Pattern I to Pattern II particulars. Maxflo Grade II is of the finest particle size, but highest permeability and lowest cake moisture content, which can be explained by an appropriate Pattern I to Pattern II particle ratio. Filtrate turbidity and % solids rejection of Grade III are not as well as the other two grades of products. A correction on turbidity may be made by switching of the pattern I to Pattern II particle ratio.

The Grade III product is a customized product for specific applications. It has the largest particle size, but lowest permeability, lowest filtrate turbidity and % solid rejection. Although there is no advantage of this product compared to Grade I and Grade II products regarding cake permeability and cake moisture, the characteristics satisfy requirements of specific applications.

Characterization		Maxflo RHA Grade I	Maxflo RHA Grade II	Maxflo RHA Grade III
pН		8.3	9.0	8.0
Particle Size, µm	d10	40	33	56
	d50	118	60	142
	d90	250	130	313
Bulk Density, lb/ft ³		15.4	16.5	14.1
Cake permeability, darcy		0.31	1.17	0.053
Filtrate Turbidity, NTU		14.2	24.5	9.6
Moisture of cake, wt	t%	65.0	55.3	66.4
% Solids Rejection*	*	99.73	99.15	99.77

Table 3. Quality Test Results on Three Grades of RHA Filter Aid Products

* Results are average values from various tests.

**5 mm precoat (formed under 15psi) followed by filtration of 0.5% ppm ISO 12103-1 A2 Fine Test Dust(Peuchot, 2000), with 0.5% RHA filter aid as a body feed.

SUMMARY

RHA filter aid products have good physical, chemical and filtration properties. Properties of RHA filter aids are highly impacted by the combustion process. The RHA product from the properly controlled suspension burning process contains high amorphous silica, low crystalline silica, and low carbon contents, which distinguish it as an excellent filter aid product.

Chemical properties, along with physical properties including particle size, shape, porous structure and particulate pattern affect filtration performance of RHA filter aid products. There are two types of particulate patterns for RHA particles. One is regular shaped porous particles; another is irregular shaped, partially porous and partially non-porous particles. A proper ratio of the two particulate patterns can be used to explain performance of different products, and can be served as a key for new product development.

Some easy to be tested physical and chemical properties such as pH, d10, d50, d90, bulk density, along with permeability, filtrate clarity, cake moisture from filtration of RHA only test, and % solid rejection from filtration of a standard fine solid suspension with RHA as precoat and bodyfeed test can be used for RHA filter aid product specification and quality monitoring.

REFERENCES

Bronzeoak Ltd, "Rice Husk Ash Market Study", ETSU U/00/0061/REP, DTI/Pub URN 03/668, 2003

Houston, D. F., "Rice Chemistry and Technology", American Association of Cereal Chemistry, St. Paul, Minnesota, 1972

Kruining, Van; Hendricus A., "Rice hull ash filter", US Patent 4765545, 1988

Li, W., C. Kiser, Q. Richard, "Development of A Filter Cake Permeability Test Methodology", American Filtration and Separations Society 2005 Topical Conference, Ann Arbor, September 2005

Peuchot, C., "Development of ISO Standards in Contamination Control and Filtration of Fluid Powder and Lubrication Systems", World Filtration Congress 8, April 3-7, 2000, Brighton, UK