Na-Bentonite and MgO Mixture as a Thickening Agent for Water-Based Paints[†]

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Abstract

Rheology plays a major role both in production and application stages of paints. Na-Bentonite, clay based thickening agent, is generally used to modify the viscosity of paints, since it is more economical and environmentally friendly compared to polymer based thickening agents. In solvent based systems bentonite is generally modified with quaternary ammonium salt to obtain appropriate polarity. However it is necessary to improve its thickening character to obtain optimum performance in water based paints. It has been reported that its flow behavior and stability can be improved by additives like MgO and LiCl for use in water based system.

In this paper, Na-Bentonite and MgO mixture was evaluated as an additive in water-borne paints. Standard paint tests such as viscosity, density, opacity, gloss, and Bucholz hardness were conducted to characterize the paint quality. It is determined that the bentonite – Mgo mixture can perform as well, or better than other thickening agents tested in this study.

Keywords: Thickening agent, Na-Bentonite, paint.

1. Introduction

Environmental regulations by EPA and European Union Commission are driving a shift from solvent-based to water-borne paints. However, it is a challenge to achieve the satisfactory service properties in water borne paints as compared to solvent based systems. Water borne paints generally show inferior properties in terms of leveling and open time compared to solvent-borne paints. This is because of relatively high evaporation rate of water and difficulty in adjusting viscosity to appropriate level. Additives are known to play a major role in determining paint service properties. Additionally, they also have considerable influence on hazardous components of paints, such as Volatile Organic Compounds (VOCs) and Alkylphenolethoxylates (APEOs). Consequently, “green” additives are being increasingly sought by

the paint industry Thickeners; for instance, represent one of the main groups of these additives.

Bentonite, which is generally used as a thickening agent in paints, is nontoxic natural material and cheaper compared to polymer based thickeners. Modification of bentonite with inorganic or organic materials converts it into a gel like structure which is appropriate for use as a thickening agent in water borne paints.

Bentonite, the commercial name for more than 80 wt% montmorillonite, is an aluminum phyllosilicate clay. Major impurities of bentonite are quartz, calcite, feldspar and biotite. Bentonites are used in a variety of diverse industries including thickeners and extenders for paints; their unique rheological properties render them to be particularly useful raw materials in foundry sand and drilling mud production^{1,2)}.

Bentonite structure can be modified to manipulate their properties. A number of studies have utilized activation and modification, using different reagents and techniques, to change the structure of bentonite³⁻⁷⁾. Activation process may involve ion exchange and ion adsorption. In modification process on the other hand, the main target is to expand the basal spacing of the bentonite by introducing larger cat-

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ions. Bentonites are generally modified to change their wettability character from hydrophilic to hydrophobic for use in solvent based applications.

Activation of bentonite with sodium increases the number of thin hexagonal laminar particles. Ion type is an important parameter, for example, Ca^{2+} ions cause “band structure” while Mg^{2+} ions cause “network structure”. Anionic clay particles serving as cation activators secure the formed networked “band structure”⁸⁾.

Thickener additives not only behave like rheology modifiers but also affect some physical properties such as gloss and hiding power of paint by modifying dispersion of primary pigments and extenders in paint formulations. Thus close attention must be paid to dispersant, thickener and pigment types to achieve optimum paint properties⁹⁾. Hydrophobically modified ethoxylated urethane [HEUR] and hydroxyethyl cellulose (HEC), which are used in water based paints have considerable effect on paint rheology. In particular different spray angles and nozzle geometries¹⁰⁾ dictate the type and amount of thickeners. The interaction of associative (HEUR type) and non-associative thickeners (HEC) with the rest of additives in paints emphasizes the role of latex particle size, surfactant type, amount and type of the thickener used in the water borne paint systems¹¹⁻¹⁴⁾. While they are very important in achieving appropriate viscosity profile across the entire shear range encountered in paint applications, they might also introduce some adverse effects. For example, associative thickener additives (HEUR) improve the open time and the leveling behavior of water-borne paints by providing an increase of network viscosity and a reduction of dispersion viscosity; they decrease the filling capacity and enhance water sensitivity problems¹⁵⁾. Paint viscosity is a crucial paint property and needs to be taken into consideration in each step of the process; production, storage and application since each step demands dramatically different viscosity values.

In this study bentonite -based thickener for water borne architectural paint was produced. Role of MgO- a cheap and less hazardous inorganic additive was investigated to achieve a gel (network) structure. Bentonite - MgO interactions were assessed by rheological and electrokinetic measurements. Performance of the optimum thickener formulation developed using bentonite - MgO mixture was compared with two commercial clay based thickeners.

2. Experimental

2.1. Materials and methods

2.1.1. Production of Na-Bentonite

TiO_2 was received from DuPont (TiPure902) and Calcite from Mississippi Corporation General properties of TiO_2 and calcite are given in **Tables 1** and **2**, respectively. Na-Bentonite used in the study was obtained from Wyoming region of the USA. Typical properties of Na-Bentonite are given in **Table 3**.

Magnesium oxide (MgO) used in the experiments was supplied by Oerac Incorporated with the properties shown in **Table 4**. Distilled and tap water were used in the production of Na-bentonite based thickener and paint, respectively. Rest of the ingredients such as dispersant, wetting agent and defoamer are commercial grade additives.

Table 1 General properties of TiO_2 pigment

TiO_2 , wt%, min.	93
Alumina	yes
Amorphous Silica	yes
Specific Gravity, g/cm ³	4.0
Bulking Value, L/kg	0.25
Organic Treatment	yes
Color CIE L *	99.6
Median Particle Size, μm	0.405
Oil Absorption, ml/100g	16.2
pH	7.9
Resistance at 30°C (86°F) (1,000 ohm)	8.1
Carbon Black Undertone	11.7

Table 2 General properties of paint grade calcite

Specific Gravity, g/cm ³	2.7
Refractive Index	1.58
Whiteness, (D 65/10) Elrepho 450 X (L, a, b)	98 ± 1
Hardness (Mohs Scale)	3
Oil Absorption, ml/100g	17 ± 2.00

Table 3 Physical properties of Na-bentonite

Screen analysis	
Dry screen, % minus 200 mesh	99.9
Dry screen, % minus 325 mesh	97.1
Wet screen, % plus 325 mesh	0.3
Slurry Properties (6% by wt. suspension)	
Viscosity, FANN [®] Viscometer 600 rpm	19
Apparent viscosity, cps	9.5
Plastic viscosity (PV)	7
Yield point, lb./100 ft ²	2
Filtrate, 30 minutes @ 100 psi, ml	15
Industrial Properties	
Moisture, %	9
Base exchange capacity meq/100 g	105
Swell index, ml	28
Plate water absorption, wt % @ 20°C /18 hr	900
Oil absorption, ml/100 g	41.3
Surface area, m ² /gram (N ₂ absorption)	20
pH, 6% suspension	9.5
Bulk density, g/cm ³ uncompacted	2.37
Bulk density, g/cm ³ compacted	3.62

Table 5 presents some physical properties of two commercial thickening agents used in water based paints, Bentone EW and Optigel CK.

Thickeners can be added both in the form of powder or paste. In this study, bentonite paste was prepared with appropriate amounts of MgO and water. In order to find out the effect of MgO dosage on the final product, 7 different mixtures of Na-Bentonite-

Table 4 General properties of MgO

Particle Size, μm	44
Median Particle Size, μm	1.29
MgO Purity	Typically 99.95%
Spectrographic Analysis	
B	<0.0001
Ca	0.0007
Cd	<0.0007
Cr	<0.0002
Cu	<0.0001
Fe	0.0006
Mn	<0.0002
Pb	<0.0001
Sr	<0.0005
Zn	<0.0006

MgO were prepared. Na-Bentonite and MgO were mixed dry and then sufficient water was added to make up 5 % solids by wt. The suspension was stirred at 6,000 rpm with a high speed “Sheen” brand laboratory disperser for 10 minutes. The suspensions were characterized by pH and viscosity profile determinations a function of time and MgO dosages. Zeta potential profile as a function of MgO dosage was also obtained for understanding the interaction of MgO with clay particles.

“Brookfield LVDV-II+ Visco-meter” with LV type spindles at 30 rpm was used for viscosity measurements. These spindles can measure fluid viscosities from 15 to 2,000,000 mPas. Zeta potential measurements were performed with “Brookhaven ZetaPlus” which measures zeta potential by Doppler shift analysis. The instrument can measure particle size rang-

Table 5 Physical properties of Bentone EW and Optigel CK

Property	Bentone EW	Optigel CK
Composition	Highly beneficiated smectite clay	Activated bentonite product
Color	Milky-white	White
Form	Soft powder	Soft powder
Density, g/cm ³	2.5	2.5
Moisture, %	10 max	8-13 max

ing from 2 nm to 30 microns. Zeta potential values were calculated using the Smoluchowski equation.

2.1.2. Production and analyses of paint with thickeners

Paint Production: Paint employed in the study was formulated according to a commercial paint recipe using ingredients listed in **Table 6**. Optimum bentonite addition established in a previous study, and two different commercial water based thickeners were separately used in this investigation. A total of 9 paints were produced with triplicates for each recipe.

Paint production process involves three main stages. At the first stage, additives such as wetting agents, dispersants, defoamer and biocides are added. The basic purpose of this stage is to provide a favorable environment for wetting and dispersion of particles. In this stage, 22.5g of additives were added to 185ml of water based on paint formula shown in **Table 6**. This mixture was stirred using a high speed stirrer for about 15minutes with 2000rpm, which is equal to a peripheral speed of 5.2m/s. In the second stage, also called as “Millbase”, pigments and extenders are dispersed in one of a number of mills depending on the type of paint to be prepared ranging from ball mills to cavitation mixers and attritors. Immediately after dispersion, the “Hegman grind” is measured as per the established standards¹⁶⁾. In our case, pigment and extenders in varying amounts, given in **Table 6**, were dispersed in the mixture formed in the first stage. The stirrer speed in the millbase stage was kept at 6000 rpm, which corresponds to a peripheral speed of 15.7m/s and the mixture was stirred for another 15minutes. During the production stage, size distributions of the paints were checked by Hegman gauge to ensure completion of the dispersion process.

In millbase, binder was not added to avoid its structural deformation under the high mechanical forces. Finally binder (370g) plus rest of the additives used in the first stage (42.5g) were mixed with 15ml water at 2000rpm that is equivalent to 5.2m/s in terms of the peripheral speed to yield a solids loading of 36.9% by weight. This stage is called “Letdown”¹⁷⁾. In this stage mixture was stirred at 2000 rpm for another 15 minutes. The final pH of the paint was measured to be 8.6. 1kg of paint was produced for each formulation. Energy losses in the mill base are minimized by adding thickeners before the dispersion stage of the production process¹⁸⁾.

Performance Evaluation: In order to evaluate the quality of the paints produced, standard paint analy-

ses such as density¹⁹⁾, viscosity²⁰⁾, opacity²¹⁾, gloss²²⁾, hardness²³⁾ and stability^{24, 25)} of the paint films were measured. The paints were applied wet onto opacity charts (15×10cm), aluminum panels (15×7.5cm) and glass panels (15×10cm) using automatic film applicator with 4-sided cylinder applicators and applicator frame. Both of them have 4 sides with different application thickness. Applicator frame and 4-sided cylinder applicators are capable of forming. 50-100-150- 200µm and 30-60-90-120µm wet film thickness respectively. Panels and charts were attached to automatic film applicator using vacuum. Application speed was kept at 150mm/s to provide sufficient accuracy.

Viscosity of paints was determined by Krebs Viscometer at a constant revolution speed of 200rpm. For opacity measurements, wet paints were applied onto an opacity chart with the wet film thickness of 200µm using 200µm side of applicator frame and automatic film applicator and cured under atmospheric conditions for two days to obtain a dry paint film. Opacity, which is one of the most important properties for both water and solvent-borne paints, was determined based on the contrast ratio of paint.

Wet paints with wet thickness of 90µm were applied on glass panels using 90µm side of 4-sided cylinder applicator and automatic film applicator and were cured for 2 days under atmospheric conditions. Gloss measurements were performed with three different incidence angles of 20°, 60° and 85°. Aluminum panels with wet paint of 200 µm in thickness were cured for 2 days under atmospheric conditions and used for determining hardness of paint. Buchholz Hardness apparatus used for determining the hardness of paint film is capable of applying 5 Newton forces.

3. Results and Discussion

3.1. Production of Na-Bentonite

All the measurements were repeated at least 3 times. Viscosity, pH and zeta potential values of bentonite as a function of seven different MgO dosages, provided in **Table 7**, indicate that the pH of the suspensions is increasing with increasing amounts of MgO up to a maximum pH of 11, and remains constant with further increase in the MgO content. Experimental fluctuation for pH, zeta potential and viscosity are ± 0.1, ± 1.5mV and ± 50cP respectively.

The variation of pH values with different amounts of MgO versus time are shown in **Fig. 1**. The measurements were taken one hour apart. Upon interacting Na-Bentonite with MgO, the pH values of the sus-

Table 6 Paint recipes with three different thickeners.

Stages	Material	Quantity, %		
		Bentonite+1.5 %MgO*	Bentone EW*	Optigel CK*
First	Water	18.5	18.5	18.5
	Antifreeze	0.8	0.8	0.8
	Biocide	0.15	0.15	0.15
	Thickener	0.2	0.2	0.2
	Dispersant	0.2	0.2	0.2
	Wetting Agent	0.3	0.3	0.3
	Defoamer	0.2	0.2	0.2
	pH adjuster	0.2	0.2	0.2
	Thickener*	0.2	0.2	0.2
Mill base	TiO ₂	28.0	28.0	28.0
	Calcite D1.7	8.5	8.5	8.5
Letdown	Styrene Acrylic Binder	37.0	37.0	37.0
	Silicone	0.1	0.1	0.1
	Film forming agent	0.8	0.8	0.8
	Synthetic thinner	1.6	1.6	1.6
	Defoamer	0.2	0.2	0.2
	Biocide	0.15	0.15	0.15
	Antibacterial	0.3	0.3	0.3
	Butyl Glycol	0.8	0.8	0.8
	Anti-settling agent	0.3	0.3	0.3
	Water	1.5	1.5	1.5
	TOTAL	100.0	100.0	100.0

pensions changed considerably in the first 5 hours and much slower thereafter indicating approaching an equilibrium.

The viscosity of Na-Bentonite suspension in the absence of MgO is 1352.5 cP. An increasing trend in viscosity with the addition of MgO was observed (see **Fig. 2**). However, at 2.5wt% MgO addition, the viscosity peaked with a value of 17126.24 cP indicating formation of a gel structure. The pH and viscosity values correlate with each other and suggest approaching an equilibrium condition in Na-Bentonite-MgO system after 5 hours.

All Bentonite+MgO suspensions yielded pseudoplastic flow behavior, i.e. their viscosity values decreased with increasing shear rate. Their viscosity also decreased with time indicating a typical thixotropic flow behavior. Such flow properties make these additives compatible to paints in terms of viscosity requirement. Viscosity of paints can be easily adjusted to desired values utilizing both pseudoplastic and thixotropic flow behavior of these additives during storage, transport and application stages.

Table 7 pH, viscosity and zeta potential values of Na-bentonite+MgO suspensions

MgO, %	pH	Viscosity, cP	Zeta Potential, mV
0.0	10.05	1352.5	-47.55
0.5	10.68	3805.1	-44.82
1.0	10.71	7496.1	-37.40
1.5	10.81	12965.0	-35.14
2.0	10.99	14998.8	-33.48
2.5	11.02	17126.2	-15.54
3.0	11.2	15987.4	-10.56

Fig. 1 Variation of pH with time for Na-Bentonite+MgO suspensions at different MgO additions.

Fig. 2 Variation of viscosity with time for Na-Bentonite+MgO suspensions at different MgO additions (LV2-30 rpm).

Zeta potential of Na-Bentonite suspensions was also studied as a function of MgO dosage. Dependence of viscosity and zeta potential on MgO addition is shown in **Fig. 3**. As expected, increasing MgO dosage renders zeta potential values more positive. It should be noted that the zeta potential of the pure Na-Bentonite was found to be -47.55 mV and increased to -10.56 mV with the addition of 3 % MgO. It is well known that Mg ions released from MgO can form MgOH^+ complexes in solution and these hydroxy complexes can adsorb onto bentonite and make it more positively charged^{3, 27}. Heterocoagulation of bentonite with MgO particles becomes conducive most probably at zeta potentials in the vicinity of -15 mV where viscosity values exceed 12,000 cP. There appears to be a critical point at which the network structure breaks down. Stability experiments of paint pastes conducted in Ishakol Paint Company laboratories revealed that upon aging high viscosity suspensions are not stable upon prolonged exposure to UV light and thus moderate viscosity levels must be maintained for achieving stable paint formulations.

pH and viscosity measurements, along with stability analysis of suspensions, reveal optimal performance at, 1.5 % MgO dosage.

3.2. Comparison of Na-Bentonite + MgO with commercial clay based thickeners

The same paint recipe was repeated three times for reproducibility of analysis. Differences among them are acceptable in terms of related standards¹⁹⁻²⁵. Viscosity, density, contrast ratio (opacity), gloss and hardness of the paint produced with different clay based thickeners are summarized in **Table 8**. Hardness is represented by indentation resistance according to the related standard.

As apparent from **Table 8**, there is no significant difference in the density of paints with different clay based thickeners. The main evaluating test for thickener effectiveness in paint is the stabilization test. After performing the physical tests on the freshly prepared paint, the remaining paint was stored (covered) in an oven at $52^\circ\text{C} \pm 2$ for 1 month. After a month, the viscosity was measured again and compared to those measured immediately after initial production. Any settling after storage was assessed. Stored paint samples were carefully mixed by hand in order to observe settling of any pigment or extender caused by agglomeration or lack of network structure.

All the paints listed in **Table 8** fall within the technical requirement range (124 ± 15 KU) in terms of

Fig. 3 Dependence of viscosity and zeta potentials on MgO dosage.

Table 8 Key physical properties of paints using different thickeners

Property	Bentonite +1.5 %MgO	Bentone EW	Optigel CK
Density, g/cm ³	1.32	1.35	1.37
Viscosity, KU Initial-Final	118.4-121.2	113.1-116.7	115.3-104.8
Contrast Ratio, %	98.2	97.65	97.2
Gloss	20°	1.5	1.5
	60°	10.65	9.15
	85°	59.40	54.80
Indentation Resistance, α B	58.8	52.6	58.8

initial viscosity values. After a month under the oven conditions ($52^{\circ}\text{C} \pm 2$), the viscosity value of paint with Optigel CK dramatically decreased while the other two paints with Bentonite+1.5%MgO and Bentone EW remained practically the same. Decrease in viscosity is an indication of pigment settling or breakdown of network structure. This is usually triggered by the lack of dispersion and agglomeration. Network structure necessary for avoiding settling of particles is provided by appropriate thickeners.

In terms of hardness (indentation resistance) value, Bentonite+1.5 % MgO and Optigel CK are almost the same and better than Bentone EW. In fact, the dominant parameters that determine the hardness value of paint are the binder type and its amount. Type of pigment and extender and amount and particle size also impact hardness. Thickeners might have an auxiliary role on hardness values through modifying particle size distribution or interaction between binder and particles.

Paints with a contrast ratio between 95% and 98% are classified as the third class paint and over 98% are considered as the first class. Therefore, the paint recipe with Bentonite+ 1.5% MgO is better than the other paints. There is no any appreciable difference among Gloss values of all the paints and all fall into the second class category.

Opacity strictly depends on dispersion of particles in the paint systems. Dispersion process has three main stages. The first one is the wetting of pigment and extender particles. In this stage, particles agglomerated due to their hydrophobic nature or resistant to wetting are made hydrophilic through with surface active agents. After the pigment particles have been partially wetted, agglomerates are broken into smaller particles by mechanical forces (high speed dissolvers, mills). Peripheral speed of a dissolver should be 18-20 m/s for millbase. Last stage of the

dispersion process is the stabilization of dispersed particles. Brownian movement results in continues collisions between the pigment particles of dispersed systems. Insufficient stabilization of the particle can therefore result in reflocculation¹⁷⁾.

Dispersion agents can modify the electric charge on the surface and introduce steric barriers that increase the stability. They can also alter the adsorption characteristics of the surface to increase the adsorption of polymeric materials or thickeners, thus increasing the barrier to flocculation¹⁸⁾. Therefore, the ability of thickeners to form a good network structure directly affects dispersion by preventing reflocculation. In this case, Bentonite + 1.5 % MgO thickener provides much higher opacity than its counterparts indicating more robust dispersion behavior.

4. Conclusions

MgO was successfully used to produce bentonite based water borne thickener from natural sources. Production of water borne paint is more complicated than solvent borne systems because more special additives such as wetting agents, dispersants and defoamers must be used to obtain at least a similar quality to the solvent one. Interaction of paint components in water based systems requires the use of an appropriate thickener type and amount to avoid any adverse effect deteriorating the paint quality. Na-Bentonite and MgO mixture performed as well or better than two commercially available clay based thickening agents. Optimal paint properties were achieved at a MgO dosage of 1.5 wt. %. Higher opacity and paint stability with Na-Bentonite and MgO mixture indicated higher resistance to reflocculation, or more robust dispersion of particles.

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Author's short biography

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Dr. Pyrgiotakis has a BS degree in Physics from the University of Crete since 1995. In 2003 he obtained his MS degree from the University of Florida, in Materials Science and Engineering with specialty on Electronic Materials. Following, in 2006 he obtained his PhD in Materials Science and Engineering with specialty on Ceramic Materials. His dissertation was dealing with the synthesis of an advanced photocatalytic particle that consist on a Carbon Nanotubes core and a titania coating. Since then he was appointed as a postdoctoral research associate at the Particle Engineering Research. His research aim is to investigate toxicity of nanoparticles and how it correlates to their properties, such size, surface morphology, shape, porosity etc. He has served as the president of KERAMOS Florida Chapter, from 2004 to 2006.

Author's short biography

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Brij Moudgil is a Distinguished Professor of Materials Science and Engineering at the University of Florida where he currently holds the Alumni Chair in Materials Science and is also the Director of the Particle Engineering Research Center. He served as the president of the Society for Mining, Metallurgy, and Exploration (SME) for the year 2006-2007. His research interests are in polymer and surfactant adsorption, dispersion and aggregation of fine particles, nanotoxicity, multifunctional nanoparticles for bioimaging, diagnosis, and therapy, nanoparticulate processing and separation technology for enhanced performance in mineral, chemical, microelectronics, pharmaceuticals, advanced materials, and resource recovery & waste disposal applications. He received his B.E degree in Metallurgical Engineering from the Indian Institute of Science, Bangalore, India and his M.S and Eng. Sc.D degrees in Mineral Engineering from the Columbia University. In 2002 he was elected as a member of the U.S National Academy of Engineering for his contributions to mineral processing and professional leadership in the particle community. He can be reached at bmoudgil@perc.ufl.edu.