New Method of Preventing Agglomeration of Urea Powder by Using Two of the Additives to Qualify it for Some Industrial Uses

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Received 28 December 2015; Accepted 28 January 2016; Available online 24 February 2016

ABSTRACT

Background: Preventing agglomeration of urea powder was made by immersing the urea powder first in anti-agglomeration solution which is soluble novolac resin with its hardener hexamethylene tetra amine (HMTA) in ethanol, and in second anti-agglomeration solution which is soluble polyvinyl alcohol (PVA) in water, the ratios of novolac resin with its hardener and PVA are (0.5-10) % by weight of the samples' weight for the two solutions. Physical properties that have been measured are the density, surface area and particle size distribution. The results showed that the best weight ratio of the first solution to reduce the agglomeration of urea is (10%), and for the second solution is (2%) by weight.

KEYWORDS: urea, antiagglomeration, PVA, novolac.

INTRODUCTION

Urea is of great importance to the agricultural industries as nitrogen – rich fertilizer; it has the highest nitrogen content available in a solid fertilizer (46 %). It dissolves readily in water. In addition to use as a fertilizer, it is used in melamine production, as an ingredient in the manufacture of resins, plastics, coatings, adhesives, textile and shrink agents, and ion – exchange resins. It is an intermediate in the manufacture of, sulfamic acid, ammonium sulfamate, and phthalocyanines [1], urea is used in detergent, cosmetics, pharmaceutics, plastic and wood industries, and it is also used as a flame retardant material [2].

Urea is obtained in two stages; the first stage is the reaction between carbon dioxide and ammonia under pressure to form ammonium carbonate (NH2 COONH4) by highly exothermic reaction. The second stage is an endothermic decomposition of the ammonium carbonate dehydration in order to convert it to urea (NH2 CONH2) [1], the dried urea density is 0.58 g/cm3.

Novolac is soluble and fusible, and requires an external curing agent, such as Hexamethylene Tetra amine (HMTA), to form the phenolic networks; its density is about 1.13–1.41 g/cm3 [3].

Polyvinyl alcohol (PVA, PVOH or PVAI) is a water – soluble synthetic polymer, it has excellent film forming, emulsifying and adhesive properties, it is also resistant to oil, grease and solvents, it has the chemical formula (C2H4O)x [4]. PVA is a resin usually used for applications that require strong binding, optical clarity, adhesion to many surfaces, toughness and flexibility. It is used in papermaking, textiles, and a variety of coatings. The excellent physical properties, chemical resistance, and biodegradability of PVA have led to the development of many commercial products. Density of PVA is about 1.27–1.31g/cm3 [5].
Literatures show that there are many studies about preventing agglomeration of powder; Maruta I. *et al.* [6] studied the addition of poly vinyl acetals (PVAL) and polyvinyl acetates (PVAC) as a preventing agent of the agglomerating of powdered urea – formaldehyde resins.


Onaka T. *et al.* [8] used a poly (oxythylene) diglycosic acid for preventing agglomeration of a powder of an amine such as piperazine and triethylenediamine powder (TEDA) which usually has coherence and adherence and thus is likely to agglomerate.

Toshio F. *et al.* [9] studied the method of fertilizer granules coating without sticking together by spraying a solution with resin, then the granules were dried with high speed hot air. The resin consists of a polyolefin and a solvent of petroleum hydrocarbon or chlorinated hydrocarbon, a solution is formed while it is hot but resin turns into Jelly's – like gel when it is cold.

In this study novolac resin and poly vinyl alcohol are used for the first time as anti-agglomeration agents for preventing urea agglomeration.

Experimental procedure:

Two materials were chosen as anti-agglomerating agents:

**A-** Iraqi novolac resin was used, it has a distinct smell and can change its structure to cross-link polymer by adding (HMTA) hardener, the latter is a white powder and the best ratio of it, is (14 %) of novolac weight [10].

**B-** Chinese poly vinyl alcohol (PVA) - 1600 was used, it is white, odorless, and the particles have bead shape.

A 100 % weight percent was used as a base weight for each sample, the first sample contained 100g of urea only. To prepare the first anti-agglomerating solutions, novolac resin and HMTA were used with weights of (2,3,4,6,8,10) g, each weight contained (86% novolac and 14% HMTA), the residue of samples weight was urea. The content of each sample of first anti-agglomeration solution is given in Table (1) and the second anti-agglomeration solution samples is given in Table (2).

### Table 1: The contents of the urea and novolac resin samples.

<table>
<thead>
<tr>
<th>HMTA (g)</th>
<th>Novolac (g)</th>
<th>Urea (g)</th>
<th>Samples' symbol</th>
<th>Samples' No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>-</td>
<td>-</td>
<td>100</td>
<td>A0</td>
<td>1</td>
</tr>
<tr>
<td>0.28</td>
<td>1.72</td>
<td>98</td>
<td>A1</td>
<td>2</td>
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<tr>
<td>0.42</td>
<td>2.58</td>
<td>97</td>
<td>A2</td>
<td>3</td>
</tr>
<tr>
<td>0.56</td>
<td>3.44</td>
<td>96</td>
<td>A3</td>
<td>4</td>
</tr>
<tr>
<td>0.84</td>
<td>5.16</td>
<td>94</td>
<td>A4</td>
<td>5</td>
</tr>
<tr>
<td>1.12</td>
<td>6.88</td>
<td>92</td>
<td>A5</td>
<td>6</td>
</tr>
<tr>
<td>1.4</td>
<td>8.6</td>
<td>90</td>
<td>A10</td>
<td>7</td>
</tr>
</tbody>
</table>

### Table 2: The contents of the urea and PVA samples.

<table>
<thead>
<tr>
<th>PVA (g)</th>
<th>Urea (g)</th>
<th>Samples' symbol</th>
<th>Samples' No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>-</td>
<td>100</td>
<td>B0</td>
<td>1</td>
</tr>
<tr>
<td>0.5</td>
<td>99.5</td>
<td>B0.5</td>
<td>2</td>
</tr>
<tr>
<td>1</td>
<td>99</td>
<td>B1</td>
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<td>3</td>
<td>97</td>
<td>B3</td>
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<tr>
<td>4</td>
<td>96</td>
<td>B4</td>
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<td>B7</td>
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<td>B9</td>
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</tr>
<tr>
<td>10</td>
<td>90</td>
<td>B10</td>
<td>12</td>
</tr>
</tbody>
</table>

Three steps are used to cover urea with the anti-agglomeration materials:

I - Mixing step:

Ethanol was put in magnetic stirrer, novolac and (HMTA) powders were mixed together by using ordinary mill, and then added to ethanol. The components were continuously mixed until novolac and (HMTA) particles were dissolved in ethanol. A calculated quantity of urea was added to with mixing the solution in each sample as shown in Table (1). The quantity of ethanol must be enough to immerse the urea, and to make a paste - like concentrated solution.
Water was put in a magnetic stirrer, its temperature was kept at 65 °C ± 5° by magnetic stirrer heater, and it was also measured by mercury thermometer, PVA was added to the water and mixed until it dissolved, then urea was added with continuous mixing, until the homogeneity was achieved.

II - Drying step:
The concentrated solutions of the first agglomeration agent were put in a oven at 60 °C to change the novolac resin structure to a cross-linked polymer and dry. All the samples of urea covered with PVA were dried at 50 °C using Germany made oven which is manufactured by Memmert Company.

III - Milling and sieving step:
All the samples obtained from step (2) were milled by using ordinary mill, and sieved by English sieve apparatus which is manufactured by Endecott Company, to obtain powder with particle size of 150 µm and less. The samples were left for six months then underwent tests.

RESULTS AND DISCUSSION

Surface area:
For measuring the surface area of some of the samples which were used in this work, Surface Area Analyzer made by Thermo Electron Corporation USA was used. The results show a significant increase in the surface area of the samples containing urea and the weight percent ratios of the novolac and PVA.

The surface area was measured for the urea powder without and with the first anti-agglomerating agent (novolac resin and its hardener), and with second anti-agglomerating agent (PVA) as given in figures (1 & 2) respectively.

![Fig. 1: The surface area of the samples containing urea and wt% novolac.](image1)

![Fig. 2: The surface area of the samples containing urea and wt% PVA.](image2)

Figure (1) shows an increase in the surface area values with an increment in amount of novolac resin but not high increment, because there is a part of novolac solute in ethanol separated as upper layer in drying step so there is a need for a mixer in the furnace for a continuous mixing but this mixer is not available, the mixing is done from time to time, so there is no chance of urea particle covered with total used novolac, the concentration of novolac resin cannot be increased more considering economic side.

The measured surface area is affected by the degree of agglomeration, so it increases when an agglomeration decreases [11].
Figure (2) shows a noticeable increase in a surface area of urea containing (2%) PVA, because of the good surface interfacial between urea particles and PVA solution which contains this weight percent ratio, the weight percent ratios of PVA are reflected on the nature of the samples, that is all the samples are brittle and can break them by hand except the sample with 2% PVA which is hard and breaks by using a mortar, therefore, this ratio can be regarded as the optimum quantity of PVA. The figure also shows sharp decreases occur in the surface area value of urea containing (10) % PVA because of the high concentration of PVA solution and the strong ability of adhesion, it binds the urea particles strongly with thick cover of which a larger part separates from urea surface during the milling step, this is in line with the method of measurement of ASTM B330 -12 [11]. Increasing the values of surface area is an index or guide to decrease in the agglomerating of urea.

**Tap density:**

Tap density also called packed density is the density of a powder, it describes the bulk density of a powder after compression by tapping the container of powder a number of times, to settle content in a container, usually a graduated glass cylinder container, under specified conditions, where bulk density of a powder describes the weight or mass of powder in a specified volume. A Tap density was measured according to ASTM B527-15 [12].

When novolac, resin is used for covering urea particles, there is a marked increase in the density, the density increment is almost linear as indicated in Figure (3), and this result is expected because novolac density is greater than the density of urea.

But when polyvinyl alcohol is used, tap density increases when the proportion of polyvinyl alcohol reaches 2% by weight, to the highest value as shown in Figure (4), it is found that the size of particulate is small when this ratio is maintained. With increasing the proportion of polyvinyl alcohol it gets particulate size increase, because of the increase resulting from the clustering effect of increasing polyvinyl alcohol. The density results are consistent with the surface area results as shown in Figure (2).

![Fig. 3: The density of the samples containing urea and wt% novolac.](image1)

![Fig. 4: The density of the samples containing urea and wt% PVA.](image2)

**Particle size:**

The device for measuring the particle size was MASTERSIZER - 3000 Malvern W.G. to measure the particle size of one sample which was (urea with 2% PVA sample) for the purpose of follow-up distribution of granular size after six months of our preparation of the sample. The result is shown as in Figure (5).
Fig. 5: The particle size distribution of urea with 2% PVA sample.

This results are in good agreement with the surface area results especially at the particle size of 100 µm for the urea particle covered with 2% PVA.

Conclusion:
It was used two methods to prevent the agglomeration of urea powder, the first solution which included the addition of urea powder with novolac resin with its hardener hexamethylene tetra amine (HMTA) by percent 14% in ethanol, and the results were good by measuring the density and surface area. The second method involved adding urea powder to polyvinyl alcohol (PVA) in water solution, and the results were good by measuring the density and surface area, especially when the ratio of polyvinyl alcohol (PVA) is 2%.

REFERENCES