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We Claim:

1. A homogenous micronutrient enriched urea composition comprising of:
   Urea having micro-nutrients and polar organic additives homogenously mixed to form a homogenous micronutrient enriched urea, the micronutrients homogenously mixed with a molten form of urea includes (0 – 25) % of inorganic salt, and wherein the amount of polar organic additives being less than 0.2% by weight of total urea weight.

2. The homogenous micronutrient enriched urea composition as claimed in claim 1, wherein said micronutrients are inorganic salts of manganese, iron, copper, zinc, cobalt, calcium, magnesium, molybdenum, potassium, boron or their mixture, wherein said salts are soluble in the molten urea.

3. The homogenous micronutrient enriched urea composition as claimed in claim 1, wherein the amount of said micronutrients is preferably less than 5% by weight of total urea weight.

4. The homogenous micronutrient enriched urea composition as claimed in claim 1, wherein said polar organic additives are polar organic compound having hydroxyl and carboxyl groups, soluble in molten urea including but not limited to oligosaccharides, polysaccharides, complex polysaccharides or their derivatives, acacia, sugar, sucrose, or Polyvinyl alcohol.

5. The micronutrient enriched urea composition as claimed in claim 1, further including a urease inhibitor composition is coated separately over the micronutrient enriched urea composition, wherein said urease inhibitor composition is extracts of neem seeds or karanja seeds.

6. The micronutrient enriched urea composition as claimed in claim 1 or 5 wherein the amount of said urease inhibitor is preferably less than 0.5% by weight of total urea composition.

7. A process for preparing a homogenous micronutrient enriched urea comprising the steps of:

   adding micronutrients and optionally a polar organic compound to molten urea under stirring condition at a temperature same or lower than the melting point of the urea; agitating the micronutrients and the optionally added polar organic compound to the molten urea for obtaining a homogenous of micronutrient enriched urea; and
   prilling the homogenous micronutrient enriched urea.

8. The process as claimed in claim 7, wherein said micronutrients are inorganic salts of manganese, iron, copper, zinc, cobalt, calcium, magnesium, molybdenum, potassium, boron or their mixture, wherein said salts are soluble in molten urea.

9. The process as claimed in claim 7, wherein said polar organic compound are selected from hydroxyl and carboxyl groups soluble in molten urea including but not limited to oligosaccharides, polysaccharides, complex polysaccharides or their derivatives, acacia, sugar, sucrose, or polyvinyl alcohol.

10. The process as claimed in claim 7, further including separately coating the micronutrient enriched urea with a urease inhibitor, and wherein the urease inhibitor is extracts of neem seeds or karanja seeds.

11. The process as claimed in claim 7, wherein the micronutrients homogenously mixed with the molten form of urea includes: (0 – 25) % of inorganic salt, and the amount of polar organic additives being less than 0.2% by weight of total urea weight.

12. The process as claimed in claim 7, wherein the step of agitating the micronutrients and to the molten urea for obtaining a homogenous of micronutrient enriched urea further includes the step of allowing cations of the micronutrients to complex with the urea, wherein certain oxides of micronutrients complex within a time period of 30 to 180 seconds.

Dated 19th Day of October, 2005



Neeti Wilson
of Ananda And Anand Advocates
Agents for the applicant

Marked Up

We Claim:

13. A homogenous micronutrient enriched urea composition comprising of:
Urea ~~enriched~~ having ~~with~~ micro-nutrients and polar organic additives homogenously mixed to form the homogenous micronutrient enriched urea~~and optionally coated with urease inhibitor composition~~, wherein the ~~maximum amount of~~ micronutrients homogenously mixed with a molten form of urea includes ~~being dependent on~~:
a. (0 – 25) % of inorganic salt,
~~a. the % solubility of inorganic salt in molten urea,~~
~~b. the % water present in urea melt and~~
~~c. the desired prill/ granule strength of urea particulates~~
and wherein the amount of polar organic additives being less than 0.2% by weight of total urea weight.

~~The micronutrient enriched urea composition as claimed in claim 1 wherein the crushing strength of particulates is increased and there is slow release of ammonium ions from urea to the soil~~.

14. The homogenous micronutrient enriched urea composition as claimed in claim 1, wherein said micronutrients are inorganic salts of manganese, iron, copper, zinc, cobalt, calcium, magnesium, molybdenum, potassium, boron or their mixture, wherein said salts are soluble in the molten urea.

15. The homogenous micronutrient enriched urea composition as claimed in claim 1, wherein the amount of said micronutrients is preferably less than 5% by weight of total urea weight.

16. The homogenous micronutrient enriched urea composition as claimed in claim 1, wherein said polar organic additives are polar organic compound having hydroxyl and carboxyl groups, soluble in molten urea including but not limited to oligosaccharides, polysaccharides, complex polysaccharides or their derivatives, acacia, sugar, sucrose, or Polyvinyl alcohol.

~~The homogenous micronutrient enriched urea composition as claimed in claim 1 or 5 wherein the total amount of polar additive is less than 0.2% of total urea~~ weight.

17. The micronutrient enriched urea composition as claimed in claim 1, further including a urease inhibitor composition is coated separately over the micronutrient enriched urea composition, wherein said urease inhibitor composition is extracts of neem seeds or karanja seeds.

18. The micronutrient enriched urea composition as claimed in claim 1 or 5~~7~~ wherein the amount of said urease inhibitor is preferably less than 0.5% by weight of total urea composition.

19. A process for preparing a homogenous micronutrient enriched urea comprising the steps of: addingdissolving micronutrients and optionally a polar organic compounds in to molten urea under stirring condition, where all of them are soluble in molten urea at a the temperature same or more lower than the melting point of the urea;
agitating the micronutrients and the optionally added polar organic compound to the molten urea for obtaining a homogenous of micronutrient enriched urea; and
prilling the homogenous micronutrient enriched urea.solidifying above solution of molten urea.; and

c. optionally coating on the surface of the particulates of above micronutrient enriched fertilizer with urease inhibitor containing coating composition.

20. A process for preparing micronutrient enriched urea as claimed in claim 9 wherein said solidification is to make prills or granules or pellets or powder of the micronutrient containing urea fertilizer.

21.20. The A process for preparing micronutrient enriched urea as claimed in claim 97, wherein said micronutrients are inorganic salts of manganese, iron, copper, zinc, cobalt, calcium, magnesium, molybdenum, potassium, boron or their mixture, wherein said salts are soluble in molten urea.

22.21. The A process for preparing micronutrient enriched urea as claimed in claim 97, wherein said polar additives are polar organic compound are selected from having hydroxyl and carboxyl groups soluble in molten urea including but not limited to oligosaccharides, polysaccharides, complex polysaccharides or their derivatives, acacia, sugar, sucrose, or polyvinyl alcohol.

23.22. The A process for preparing micronutrient enriched urea as claimed in claim 9 7, further including wherein said separately coating the micronutrient enriched urea with a urease inhibitor, and wherein the urease inhibitor is extracts of neem seeds or karanja seeds.

24.23. A The process for preparing micronutrient enriched urea as claimed in claim 97, wherein the micronutrients homogenously mixed with the molten form of urea includes: (0 – 25) % of inorganic salt, and the amount of polar organic additives being less than 0.2% by weight of total urea weightamount of said micronutrients depends upon
a. the % solubility of inorganic salt in molten urea,
b. the % water present in urea melt and
c. the desired prill/ granule strength of urea particulates.

25.24. The process as claimed in claim 7, wherein the step of agitating the micronutrients and to the molten urea for obtaining a homogenous of micronutrient enriched urea further includes the step of allowing cations of the micronutrients to complex with the urea, wherein certain oxides of micronutrients complex within a time period of 30 to 180 seconds.

~~26.~~25. ~~A micronutrient enriched urea composition and method of preparing the same substantially as herein described with reference to accompanying examples and drawings~~.

Field of the invention: This invention relates to micronutrient enriched urea fertilizer compositions and methods of preparing them. More particularly, this invention relates to urea-micronutrient particles having micronutrients homogeneously dispersed throughout the urea particles and to a process of preparing such particles.

Background of Invention

All plants require a balanced supply of micronutrients for its growth. Therefore farmer needs balanced nutrient fertiliser with minimum cost. The type of nutrient content in balanced fertiliser depends on type of plant, types of soil and weather. As soil, climate and agricultural systems changes region to region therefore it is preferable to manufacture balance fertiliser product near to its end use.

Among all the nutrients required for plant growth, plant takes carbon, hydrogen, oxygen from water and air, while nitrogen, phosphorous, potassium, sulphur, calcium, magnesium are taken as macronutrients and zinc, copper, iron, manganese, boron, molybdenum, chlorine, cobalt as micronutrient either from soil or from fertiliser.

Nitrogen is one of the primary nutrients required by plants for robust growth. Most fertilizer compositions include nitrogen, most commonly in the form of urea. Urea applied to soil is enzymatically hydrolyzed to ammonia by an enzyme known as urease, which is found in soil microorganisms. However, this conversion is very rapid and exceeds the rate at which plants can utilize the released ammonia. The excess ammonia is either further converted to nitrites and nitrates by nitrifying bacteria in the soil (including Nitrosomonas and Nitrobacter species), which leaches into the water, or is lost to the atmosphere by denitrification, as known in the art. However, only about 40% to 50% of urea applied in conventional fertilizers appears to be utilized by plants, while the remainder is lost by the above-described processes. Obviously, a considerable amount of applied urea is wasted, as plants do not utilize it.

Soil and climate factors influence ammonia loss. Low humidity, high temperature, low clay & organic matter content in soil and high soil pH favors ammonia loss. Use of ecofriendly urease inhibitor like neem seed extract and/or karanja seed extract reduce ammonia loss from urea fertilizer.

All most all plants require nitrogen in more quantity compared to other nutrients. Among all nitrogen fertilizers urea is the major one. Therefore it is preferable to supply other nutrients with urea fertilizer. Such nutrients may be added with urea granules with the help of binder. For keeping its cost low, the binder amount should be just sufficient to bind nutrients on the surface of urea prills.

In the fertilizer industry, there are methods available for coating granules with particular additives. Most of methods involve a chemical reaction between the chemical compounds in the granules to be coated and use additives to affix the granules. Oil and wax have also been used to stick nutrients. Other methods for coating granules involve using binders to attach nutrients to fertilizer granules.

The methods for adding zinc salts to fertilizer compositions for one-step application include dry blending, coating, and solution dispersion methods. Two major disadvantages detract from all of these methods, viz., hygroscopicity and the difficulty of obtaining a uniform product.

U.S. Patent 3353949 discloses a method for preparing a coated granular fertilizer wherein the finely divided zinc nutrient particles are mixed with the granular fertilizer and an aqueous solution of conditioner is added and mixed until adhesion of the nutrient to the fertilizer is attained.

Dry blending of zinc salts with particulate fertilizer compositions is generally unsatisfactory, since combinations of zinc salts and urea are usually hygroscopic and thus absorb moisture from the air and agglomerate into an unusable solid cake. Furthermore, powdery zinc compounds readily separate from the

particulate material, resulting a waste of zinc, often interfering with the proper operation of fertilizer dispensing machinery, and causing non-uniform application to crops.

In one attempt to overcome such disadvantages, United States Patent 3981713 discloses a particulate fertilizer comprising urea particles having a fertilizing quantity of available zinc in the form of zinc oxide which is homogeneously dispersed throughout the urea particles. The urea-zinc oxide particles may be prepared by dissolving zinc oxide into molten urea containing less than 12% by weight water, based on water/urea + water, and solidifying the molten urea-zinc oxide blend into particles of any desired form.

This process has however suffers from following disadvantages:
- prills containing higher amounts of zinc oxide as an internal additive are softer than normal urea prills;
- the particulate urea-zinc oxide products of this process are more hygroscopic than urea; thus
- the caking tendency increases on storage in high humid environment.

The instant invention seeks to overcome the above problems by providing a particulate urea- solid micronutrients fertilizer having improved prill strength. The novel process disclosed includes the step of addition of polar compounds to the molten urea along with micronutrients to attain high prill strength.

SUMMARY OF INVENTION:

The object of the present is to provide urea- solid micronutrients fertilizer wherein the solid micronutrients are homogeneously dispersed throughout the urea prills. In cases where micronutrients are water insoluble but soluble in molten urea, salts are delivered to the soil as fine particulates, which is more effective than their coarse particulates.

The instant invention provides a method of preparing a chemical composition, from which micronutrients can be delivered with urea prills having low rate of ammonium ion release from urea to the soil.

It is a further object of the invention to provide micronutrient enriched urea fertilizer coated with urease inhibitor like extracts of neem seed or karanja seed, to enable release of ammonium ion in a slow rate and low initial soil pH. This increases the availability of ammonium ion in soil for longer time in a sustained manner and the availability of ammonium ion to the plant also increases.

In view of the above the micronutrient-enriched urea is produced by using the following method:

Detailed description of the invention

The instant invention provides for micronutrient-enriched urea produce by dissolving micronutrients and polar organic compounds in molten urea.

The micronutrients used by plants and used in the instant invention comprise of inorganic salts of manganese, iron, cupper, zinc, cobalt, calcium, magnesium, molybdenum, potassium, boron or their mixture, which are soluble in molten urea. These salts undergo reaction with urea and form complexes.

In the case of Zinc Oxide being used, Zinc oxide like most of other oxides undergo reaction as follows

$$2*NH2CONH2 + ZnO \text{ --------> } Zn\ (NHCONH2)2 + H2O$$

Water is liberated in the reaction. Presence of water decreases the prill strength and increases prill caking tendency. Therefore it is preferable to start

prilling/solidifying from low water content micronutrient containing molten urea solution.

In presence of water, zinc hydroxide gets precipitated as fines from zinc oxide micronutrient enriched urea particulates. Due to smaller particle size and more water solubility of zinc hydroxide, the zinc ion availability in soil for plant increases compared to zinc oxide added as such to soil or by coating on urea surface.

In presence of other inorganic compounds reaction may be different . As in the case of ZnSO4 the reaction undergoes as follows:

$$2*NH_2CONH_2 + ZnSO_4 \longrightarrow Zn(NHCONH_2)_2 + H_2SO_4$$

$$NH_2CONH_2 + H_2SO_4 \longrightarrow (NH_2CONH_2)H_2SO_4$$

Sulphuric acid generated is corrosive. Therefore addition of more amount of zinc sulphate is not preferred.

Similarly in case of inorganic salts containing chlorides, nitrates or nitrites, corresponding acids will be generated, which are also corrosive and special precaution is required.

The maximum percentage of micronutrients that can be incorporated in making micronutrient containing urea fertiliser depends on solubility of that inorganic salt in molten urea.

Prill strength depends on the amount of micronutrient used, type of organic salt used, moisture content of the fertilizer,

If micronutrient amount is high then prill strength decreases. The prill strength of some of zinc salt enriched urea fertiliser without adding any organic compound to molten urea are given in Table 1.

Table 1

Compounds	Statistical average for prill strength(in Kg) after 3 days of preparation for prills having approximately 4mm diameter
Urea prills	2.2
5% Zinc oxide containing urea prill	3.3
15% Zinc oxide containing urea prill	1.8
25% Zinc oxide containing urea prill	0.2
5%Zinc carbonate containing urea prill	2.1
10%Zinc carbonate containing urea prill	1.0

Similarly, prill strength depends upon the type of inorganic salt is exemplied by measuring the for prill strength of different zinc salts enriched urea fertiliser depicted in Table 2.

Table 2

Compounds	Statistical average for prill strength(in Kg) after 3 days of preparation for prills having approximately 4mm diameter
Urea	2.20
5% Zinc oxide containing urea	3.3
5%Zinc carbonate containing urea	2.10
5%Zinc chloride containing urea	1.9
5%Zinc sulfate containing urea	2.0
5%Zinc nitrate containing urea	1.7

It has been similarly observed that the Prill strength decreases with increase in moisture content in fertiliser composition. Therefore it is clear that the % of micronutrient that can be delivered with urea prills mainly depends on (a) solubility of the micronutrient in molten urea (b) Prill or granule strength required.

The most preferred embodiment of the invention included polar organic compounds in the micronutrient enriched urea composition. The polar organic compound in may be selected from oligosaccharides, polysaccharides, complex polysaccharides or their derivatives, more particularly acacia, Sugar(sucrose) and /or Polyvinyl alcohol . These compounds have polar groups, which undergo reaction with urea to impart strength to particulates.

The total amount of polar organic compounds used is more preferably less than 0.1% of total urea. The higher doses may face some difficulties such as

- foaming may occur at the time of dissolving organic additives and
- water or acid content in composition may increase, which would lead to urea prills or granules having lower crushing strength.

It is one of the preferred embodiments of the invention, the micronutrient enriched urea fertilizer surface is coated by a coating composition which contains urease inhibitor such as extracts of neem seeds, karanja seeds, etc. The coating is used in an amount preferably less than 0.5% (by wt) of total fertilizer composition. This coating is made to increase the availability of ammonium ion to soil by decreasing pH of the soil and deactivating urease enzyme.

A typical process of preparing neem seed extract coated 5% zinc oxide enriched urea fertilizer comprises of following steps:

5gm of micronutrient salt such as zinc oxide is added to 95 gm of molten urea at 135-140 degC (above melting point of urea) and stirred till it is completely soluble. Thereafter 50 ppm of organic compound such as sugar (sucrose) is added to it. Water is removed from the system to less than 0.4 ppm. Solublisation of Zinc oxide and organic compound in molten urea gives clear, transparent solution. The above hot liquid is passed through a prilling apparatus. The inside

temperature of prilling apparatus should be higher than freezing point of zinc salt solublised molten urea. Urea prills are formed by allowing molten urea droplets to descend in aliphatic oil having viscosity of 10 to 40 poise. After solidifying, the prills are taken from the oil and washed with organic solvent like chloroform or petroleum ether to remove the oil.

Solidified micronutrient containing urea prills prepared as per above process is mixed with 0.1 gm of neem seed extract. It may then be stored.

The invention is hereinafter described in detail with the help of different examples of the process steps used to obtain the micronutrient-enriched urea.

Example 1

The micronutrients when soluble in molten urea at the temperature same or more than melting point of urea, the polar organic compounds or their mixture are added as such or as their aqueous solution. The solution of micronutrient in molten urea is then solidified in appropriate apparatus as prills or granules or pellets or powder-form to obtain the micronutrient enriched urea fertilizer.

The freezing temperature as well as melting temperature becomes lower with addition of inorganic salt (micronutrients) to urea compared to normal urea. Approximate lowering of freezing temperature for some of the compositions are provided in Table 3 below:

Table 3

Fertilizer Composition	Freezing point (deg C)
2.5% Zinc oxide containing urea	131
7.5% Zinc oxide containing urea	120
10% Zinc oxide containing urea	119
15% Zinc oxide containing urea	118
10% Zinc carbonate containing urea	128
5% Zinc sulphate containing urea	130

10% Zinc sulphate containing urea	122
Urea	133

Example 2:

The micronutrient enriched urea fertilizer surface is coated by a coating composition which contains urease inhibitor such as extracts of neem seeds, karanja seeds, etc. The coating is used in an amount preferably less than 0.5% (by wt) of total fertilizer composition. This coating is made to increase the availability of ammonium ion to soil by decreasing pH of the soil and deactivating urease enzyme. Table 4 presents the various compositions with and without micronutrient as well as the coating in case of zinc oxide being used as the micronutrient to be added in the urea.

Table 4

	Urea having polyvinyl alcohol and 10% Zinc oxide coated with neem seed extract	**Urea having polyvinyl alcohol and 10% Zinc oxide**	**Normal urea**
Neem seed extract used in coating of urea(gm)	0.01	0	0
Urea (gm)	1	1	1
Polyvinyl alcohol(gm)	200ppm	200ppm	0
Zinc oxide	0.1	0.1	
Filtrate of mixture* of urease	5	5	5
Distilled water (gm)	93.99	93.99	94
Total wt (gm)	100	100	100

**(jack bean meal) and soil in distilled water (gm)*

In one of the embodiment of the invention oil is sprayed on the surface of micronutrient enriched urea fertilizer particulates.

Example 3

The pH of the fertilizer solution was monitored at different interval of time in the filtrate of aqueous solution of soil containing urease inhibitor. It is observed that if soil pH is lower, the ammonium ion loss from the soil is less. Table 5 shows the pH measured in the solution at different time intervals. The same data has been depicted in Figure 1 to clearly visualize the impact of addition of micronutrients and coated micronutrients in the Urea pH.

It is clearly seen (Figure 1) that due to coating of neem seed extract on urea prills, the pH of fertiliser solution is less compared to non-coated micronutrient containing urea. The pH of normal urea is still low compared to other two fertilizer (Figure 1). This may be due to the presence of 10% Zinc oxide in other fertilizers.

Table 5

Time	Urea having polyvinyl alcohol and 10% Zinc oxide coated with neem seed extract	Urea having polyvinyl alcohol and 10% Zinc oxide	Urea
0hr	7.80	8.16	7.10
2hr	7.91	8.25	7.35
4hr	7.93	8.35	7.60
6hr	7.95	8.45	7.71
8hr	7.97	8.50	7.75

Example 4

The Prill strength for some of the compositions used in the instant inventions are provided in Table 6, 7 and 8.

It may be observed in Table 6 that the addition of different additives such as pentaerithritol, polyvinyl alcohol or sugar (sucrose) increases the strength of zinc oxide containing urea prills.

Table 6

Composition	Additive	*Statistical average for prill strength(in Kg)by crushing prills having different diameter*		
	50 ppm	1mm	2mm	3mm
Urea	-	0.29	0.58	1.2
11% zinc oxide with urea	-	0.22	0.45	0.95
11% zinc oxide with urea	Pentaerithritol	0.582	1.266	2.098
11% zinc oxide with urea	Sugar(sucrose)	0.434	1.003	1.578
11% zinc oxide with urea	Polyvinyl alcohol	0.56	1.093	1.39
11% zinc oxide with urea	Acacia	0.328	0.546	1.036

Table 7 shows that prill strength depends on the amount of additive added .It is not necessary always that the prill strength will increase with increase in binder content. This may be attributed to the foaming or more water content in molten urea composition.

Table 7

Composition				
		Statistical average for prill strength(in Kg)by crushing prills having different diameter		
	Polyvinyl alcohol	1mm	2mm	3mm
Normal urea		0.29	0.58	1.2
11% zinc oxide with urea		0.22	0.45	0.95
11% zinc oxide with urea	1000ppm	0.37	0.85	1.17
11% zinc oxide with urea	500ppm	0.64	1.3	2.2
11% zinc oxide with urea	50ppm	0.582	1.266	2.098

The effect of the additives used in different combinations is presented in Table 8. The prill strength on addition of combination of Pentaerithritol, polyvinyl alcohol, sugar(sucrose) increases for a particular combination and also decreases for another combination. It is therefore required to choose right combination.

Table 8

UREA +	*Additive*				*Statistical average for prill strength(in Kg)by crushing prills having different diameter*		
Zinc Oxide	**Pentaerithritol**	**Sugar (sucrose)**	**Poly vinyl alcohol**	**Acacia**	**1mm**	**2mm**	**3mm**
0	-	-	-	-	0.29	0.58	1.2
11%	-	-	-	-	0.22	0.45	0.95
11%	-	-	50ppm	-	0.582	1.266	2.098
11%	5ppm	-	50ppm	-	0.59	1.67	2.8
11%	-	5ppm	50ppm	-	0.7	1.7	3
11%	-	-	50ppm	5ppm	0.588	1.35	2.35
11%	25ppm	-	50ppm	-	0.57	0.794	1.09
11%	-	-	50ppm	25ppm	0.552	0.67	1.112
11%	-	25ppm	50ppm	-	0.345	0.643	0.985

Example 5

One of the preferred embodiment of the invention uses Zinc Sulphate in an amount of 2% by weight in the urea fertiliser along with polyvinyl alcohol. Table 9 reveals that the prills strength increases even for 2% zinc sulphate enriched urea fertilizer on addition of the additive.

Table 9

Composition	*Additive*	*Statistical average for prill strength (in Kg) by crushing prills having different diameter*		
UREA +	**Polyvinyl alcohol**	**1mm**	**2mm**	**3mm**
2% zinc sulphate mono hydrate		0.5	0.93	1.122
2% zinc sulphate mono hydrate	75ppm	0.685	1.234	1.955

ABSTRACT

MICRONUTRIENT ENRICHED UREA

A homogenous micronutrient enriched urea composition comprising urea having micro-nutrients and polar organic additives homogenously mixed to form a homogenous micronutrient enriched urea and a process for preparing such homogenous micronutrient enriched urea.

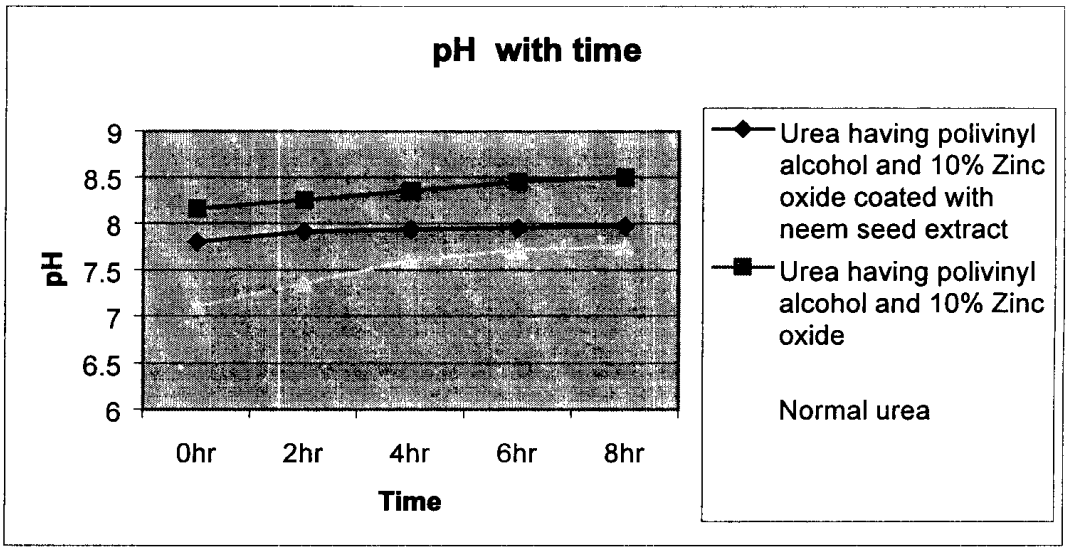


Figure 1

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