Use of treated cement kiln dust as a nano material partially replaced in cement mortars

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Abstract: Cement kiln dust (CKD) is a by-product material of cement manufacturing. The composition of this material is approximately like the cement composition but it carries high ratio of alkali which is harmful material if it is used in structural construction members. In this study it was used after removing most of alkali by washing, in cement mortars as partial replacement of cement. After washing it was left to be dried. The material was grinded by Los Angeles machine firstly and then milled by a planetary mill in periods of 1, 1.5, 2, 2.5, 3, 3.5, and 4 hours respectively to reach nano sizes of particles. It was added to cement mortars using five ratios, 0% (without milling as control ratio) and an addition of 5%, 10%, 15%, and 20% ratios compared to cement weight respectively. The properties which were studied include compressive strength of mortar through curing periods 7, 28, 56 days, physical and chemical properties of the mortars. The results of this work ensure the work of one of the authors in the literature and an improvement in mortar compressive strength reaches 35%. Other physical and chemical properties were also improved. Depending upon the governed results washed CKD can be used safely in cement mortars.

1. Introduction
The most innovative developments nowadays which make revolution in industries is the nanotechnology especially in material science. It deals with sizes of particles in a range from 1 to 100 nm. This change in particle size possess new properties as their surface area enlarged in relation to their volume so-called "size-induced functionalities" (1). Nanoparticles, i.e. particles with a diameter of less than 100 nm, have been the object of scientific research for many years now. Ultrafine particles are, for example, harder and more break-resistant than larger particles. One of the major media to use nanotechnology is the by-product cement kiln dust (CKD). This material forms during the high heating and burning released in the cement kiln. The cement dust will appear in the upper part of the kiln and it was discharged through hydrostatic precipitators. CKD is very harmful material due to the following reasons:

1. The high cost needed to raise this material and transport it outside the factory.
2. High quantities are produced daily reaches about 250 ton as an average discharge, so this needs efforts in so many sides like place of stocking, maintenance of precipitators and other equipments.

3. Healthy effect on human being, animals, plants and ground if this material discharged through chimneys.

4. It has high alkali compound therefore it needs certain treatments to be reused as a benefit material.

So many efforts had been done to fulfill that aim (as beneficial material). A summary of such efforts will be mentioned here:

1. It was tested and used in manufacturing cement bricks as a partial replacement of cement material by Hakim S. M. and Radi M. Z [2].
2. As partial replacement; as a filler in asphalt concrete; and in other uses such as contaminants, filtration [3, 4, 5, 6 and 7].
3. Studies investigated the possibility of using cement kiln dust in concrete mixes (8, 9, 10, 11, 12, 13 and 14).
4. Study of returning the cement kiln dust to the cement manufacturing process by washing it so many times to remove harmful alkali material [15].
5. Studies including the change of particle size of CKD as nano particles. Alnahhal W. et al., (16) studied the effect of using CKD as nano-material on the strength of cement mortars. They concluded that the strength of the mortar increased by about 15-30% when the dust was milled 4 hours by a planetary mill and added to the mixture by a 20% ratio.

The present paper makes use of the paper presented by Alnahhal W. et al. to use CKD after removing the alkali harmful compound from it; i.e. improving the characteristics of composition of the cement kiln dust. It will follow the same procedure done by him but with using un-milled and milled treated (washed CKD) throughout whole tests.

2. Experimental work

The experimental work comprises casting 4X7 mortar mixtures. The first three cubes are the control cubes where the ratio of CKD is zero. The added ratios of CKD start with % as percentage ratio with respect to cement weight. Each mix was represented by three cubes.

2.1. Experimental program

Material properties: The materials used in this work were cement, sand and washed CDK. The cement used is an ordinary Portland cement type 1 which was taken from Kufa cement factory in the middle region of Iraq. This material was conformed with Iraqi specifications (IQ.S. No.5/1984). The chemical composition of cement and its physical analysis properties can be seen in tables (1 and 2) respectively.

| Table 1. Chemical composition of cement. |
|----------------------------------------|
| Item | Chemical composition | Weight % | Iraqi specifications No.5/1984 |
|------|----------------------|----------|-----------------------------|
| 1    | Lime CaO             | 62.38    | --                          |
| 2    | Silica SiO₂           | 20.53    | --                          |
| 3    | Alumina Al₂O₃        | 4.39     | --                          |
| 4    | Iron Oxide Fe₂O₃     | 4.37     | --                          |
| 5    | Magnesia MgO         | 2.90     | ≤5%                         |
| 6    | Sulfate SO₃          | 2.32     | ≤2.8%                       |
| 7    | Loss on Ignition L.O.I.| 2.34   | ≤4%                         |
| 8    | Insoluble Residue I.R.| 0.70  | ≤1.5%                       |
| 9    | Lime Saturation Factor L.S.F.| 0.93 | 0.66-1.02                   |
| 10   | Tri-Calcium Aluminates C₃A | 7.12 | --                          |
Table 2. Physical analysis of cement.

| Physical property                                      | Test results | Iraqi specifications No.5 /1984 |
|--------------------------------------------------------|--------------|----------------------------------|
| Fineness by air permeability method (Blain)-cm²/gm     | 2997         | 2300≥                            |
| Setting time using Vicate’s instrument                  |              |                                  |
| Initial (min.)                                         | 161          | 45≥                              |
| Final (min.)                                           | 226          | ≤600                             |
| Compressive strength for cement paste in N/mm²         |              |                                  |
| 3 days age                                             | 31.60        | 15≥                              |
| 7 days age                                             | 38.7         | 23≥                              |

2.1.1. Fine aggregate:
A natural sand brought from Al-Najaf region in Iraq was utilized as fine aggregate for the mortar mix. The maximum size of sand was (4.75 mm) with smooth texture and rounded particle shape. This material was conformed with Iraqi specifications (IQ.S. No.45/1984) zone-III. The specific gravity was 2.65, sulfate content SO₃ is 0.21 which is less than permissible ratio 0.5 as recommended by Iraqi specifications (IQ.S. No.45/1984).

2.1.2. Cement Kiln Dust (CKD): This material is a by-product fine grained and highly alkaline. The material was collected directly from the electrostatic precipitators in Kufa cement plant. To use this material as a nano additive to the cement it must pass through the following steps to be ready before using it:
   1. Collecting the material.
   2. Testing it by chemical analysis.
   3. Washing the dust to remove soluble alkali.
   4. Removing alkali and drying the residue material.
   5. Testing the residue material.6. Milling this residue so many times to reach the nano sizes.

The material was collected directly from the stock of the hydrostatic precipitators mouth. The method used is like that consumed by aggregate sampling as stated by ASTM D75 / D75M – 14 [17]. The selected sample was taken to the laboratory and analyzed to find out its composition as in table 3. The degree of solubility of alkali can be seen in table 4 which was measured in 18 °C.

Table 3. Chemical analysis of cement dust

| Compound       | Percentage ratio of compound in dust |
|----------------|--------------------------------------|
| Silica SiO₂    | 15.46                                |
| Alumina Al₂O₃  | 3.91                                 |
| Iron Oxide Fe₂O₃ | 3.05                               |
| Lime CaO       | 43.40                                |
| Magnesia MgO   | 2.98                                 |
| Sulfate SO₃    | 6.34                                 |
| Potassa K₂O    | 2.44                                 |
| Soda Na₂O      | 1.42                                 |
| Chloride Cl    | 0.29                                 |
Loss on Ignition L.O.I.  28.86  
Total  100.15  
Free lime  2.96  

| Alkali            | Degree of solubility Gram/100 ml in 18 °C |
|-------------------|------------------------------------------|
| Clayey and chloride | KCl                                     32.95 |
|                   | NaCl                                    35.85 |
|                   | CaCl₂                                   33.19 |
|                   | MgCl₂                                   55.81 |
| Clayey and Sulphates | K₂SO₄                               11.11 |
|                   | Na₂SO₄                                 16.83 |
|                   | CaSO₄                                   0.20 |
|                   | MgSO₄                                   35.43 |
| Clayey and Carbonates | K₂CO₃                               108.00 |
|                   | Na₂CO₃                                 19.39 |
|                   | CaCO₃                                   0.0013 |
|                   | MgCO₃                                   0.10 |

Table 4. Degree of solubility of alkali Compounds.

The dust was mixed with a water using ratio of 55% by weight of water to dust. This process took about one hour time to get homogeneous mix. It was noticed that the solution of the dust with water is alkaline having the following properties:

1. The pH is between 12 to 13.
2. It was seen that sulphate, chlorides and clayey alkalis are soluble in water as it shown in Table 4. The degree of solubility increases proportionally with heat increase. All of the compounds have high degree of solubility except for the calcium sulphate.
3. When the mix solution was left for more than one hour the dust sedimented downwards. Then it will be separated from the solution for more than two hours gradually and depending on this property the alkalis salts can be separated from the sedimented dust.

This process is ended by removing alkalis from the solution and the addition of water can be repeated several times to remove whole alkalis. In the lab it was repeated three times. The residual which is precipitated at the base of the mixing container was dried by air and heat. The residue material after drying was analyzed to find out the ratios of constituents. The analysis test was done and the results can be seen in Table 5 below.

Table 5. Chemical analysis of cement dust before and after washing.

| Compound     | Percentage ratio of compound in dust before washing | Percentage ratio of compound in dust after washing |
|--------------|-----------------------------------------------------|--------------------------------------------------|
| Silica SiO₂  | 15.46                                               | 16.02                                            |
| Alumina Al₂O₃| 3.91                                                | 3.77                                             |
| Iron Oxide Fe₂O₃| 3.05                                   | 3.49                                             |
| Lime CaO     | 43.40                                               | 44.45                                            |
| Magnesia MgO | 2.98                                                | 3.03                                             |
| Sulfate SO₃ | 6.34                                                | 5.50                                             |
| Potassa K₂O  | 2.44                                                | 1.14                                             |
| Soda Na₂O    | 1.42                                                | 0.84                                             |
| Chloride Cl  | 0.29                                                | 0.247                                            |
| Loss on Ignition L.O.I. | 28.86               | 22.11                                            |
| Total        | 100.15                                              | 100.60                                           |
| Free lime    | 2.96                                                | 0.20                                             |

alkali is the sodium alkali equal to Na₂O+0.65 Thus, total before washing it was 3.006 and washing it was 1.581 very good result. The
step in this procedure to reach the nano sizes in a range of $10^{-9}$ in particle diameters is to grind the residue and then collecting the material to be placed in a milling machine container. The grinding was achieved by Los-Angeles machine that is used in the abrasion test of coarse aggregates as shown in photo 1. The container has three rotary steel blades fixed to its body and Cast iron or steel balls, approximately 48 mm in diameter and each weighing between 390 to 445 g; twelve balls are required. The principle of Los Angeles here is to make grinding action by use of standard steel balls which when mixed with dust and rotated in a drum for specific number of revolutions changes the dust into very fine powder to help the planetary ball mill in its task as seen in photo 2.

Photo 1. Los- Angeles machine

Photo 2. Planetary Ball Mill from RETSCH

2.2. Planetary Ball Mills

In the planetary ball mill, every grinding jar represents a planet”. This planet is sun wheel. When the sun wheel every grinding jar rotates around its own axis, but in the opposite direction. Thus, centrifugal and Coriolis forces are activated, leading to a rapid acceleration of the grinding balls figure 1. The result is very high pulverization energy allowing for the production of very fine particles. The enormous acceleration of the grinding balls from one wall of the jar to the other produces a strong impact effect on the sample material and leads to additional grinding effects through friction. For colloidal grinding and most other applications, the ratio between the speed of the sun wheel and the speed of the grinding jar is $(1: -2)$. This means that during one rotation of the sun wheel, the grinding jars rotate twice in the opposite direction.

Figure 1. In the planetary ball mill, centrifugal and Coriolis forces permit grindings down to the submicron range
Same procedures followed by Alnahhal W. et al. [16], will be reused in milling the dust and it is good study to compare the current work results with. The following steps were done to reach the nano scale sizes:

1- Further grinding, mixing and homogenization of dust were done by planetary ball mill called Pulverisette-5 to produce ultra-fine materials as the nano-scale.

2- The grinding jar which was used in this study with a size of 250 ml and ten grinding balls of diameter 30 mm were placed inside the jar.

3- A ratio of (0.5-0.75) between CKD mass with respect to grinding balls mass were maintained throughout the milling process.

4- 200 rev./min. was the speed of the supporting disc.

5- The CKD was milled for seven different milling times (1, 1.5, 2, 2.5, 3, 3.5, and 4 hours).

6- It was observed that when a certain degree of finenesses is achieved, the agglomeration of the particles tended to increase. This effect can be of decisive importance, even with a grain size of less than 10 microns. As a result, the individual particles began to stick together to the grinding balls and to the inner wall of the bowls. This agglomeration might increase due to any further milling.

2.3. Cement mortar sample

70 X 70 X 70 mm cubes were used to prepare the mortar samples to be tested and a total of 28 cubes were molded taking into consideration that 3 of them are without CKD added to be considered as control cubes. Four different percentages of nano-CKD (5, 10, 15, and 20%) were added to the cement sand mix at seven different milling times (1, 1.5, 2, 2.5, 3, 3.5, and 4 hours). Same procedure followed by Alnahhal W. et al. [16], will be used in mortar mix to facilitate the comparison of the results. The mix proportions of mortars are as follows: Water weight is kept constant and equal to one Kg or one liter also sand kept unchangeable and equal to six Kg whereas cement and the quantity of CKD added are the variable parameters. The water-to-binder ratio (w/b) is also kept unchanged and equal to 50%. The amounts of cement and different ratios of CKD added can be seen in Table 6. Materials were mixed in accordance with ASTM C192M-16 (ASTM C192M 2016). The compressive strength of all cement mortar cubes was measured according to the provisions of ASTM C109M-16 (ASTM C109M 2016). For each mixture three samples were tested. The results after 7, 28, and 56 days of curing, and their average were reported as the compressive strength of the specimens.

Table 6. Weight Proportions of the Cement Mortar Ingredients.

| Mixture Type ( %Ratio of CKD added as compared to weight of cement used) | Cement (g) | Nano-CKD (g) added to the mix |
|---------------------------------------------------------------|------------|------------------------------|
| OPC (0% CKD)                                                  | 2,000      | -                            |
| CKD (5%)                                                      | 1,900      | 100                          |
| CKD (10%)                                                     | 1,800      | 200                          |
| CKD (15%)                                                     | 1,700      | 300                          |
| CKD (20%)                                                     | 1,600      | 400                          |

3. Results and Discussion

The results of this study focus on the most important factors which are physical properties, chemical properties besides the compressive strength of the cement mortar.

3.1. Physical properties of the un-milled and milled cement kiln dusts.

The physical properties of the CKD milled or un-milled are including the specific gravity, particle size, surface area and initial setting time. These characteristics of mortar are very important in early understood of the behavior of the compressive strength of the mortar. The variation of these
parameters reflects on the behavior of compressive strength as follows:
Increasing of specific gravity and surface area means compressive strength developments. Decreasing in particle size means increase in surface area and that will lead to increasing in strength. Finally the decrease in setting time means quick reaction is gained and that is needed to reach an early compressive strength especially in cement mortar works. The results of this test depending on the time of milling were mentioned in Table 7.

Table 7. Physical properties of CKD during each milling time.

| Material                | Test Time | Specific Gravity | Particle Size | Surface Area ($cm^2/g$) | Initial Setting Time (min.) |
|-------------------------|-----------|------------------|---------------|-------------------------|-----------------------------|
| CKD (un-milled)         |           | 2.58             | 200-100 nm    | 3,890                   | 152                         |
| 1 hr CKD milling        |           | 2.62             | 100 nm        | 8,900                   | 145                         |
| 1.5 hr CKD milling      |           | 2.68             | 100 nm        | 9,898                   | 138                         |
| 2 hr CKD milling        |           | 2.71             | 100-50 nm     | 14,150                  | 129                         |
| 2.5 hr CKD milling      |           | 2.76             | 100-50 nm     | 15,750                  | 124                         |
| 3 hr CKD milling        |           | 2.79             | 50 nm         | 16,990                  | 120                         |
| 3.5 hr CKD milling      |           | 2.81             | 50-20 nm      | 17,600                  | 117                         |
| 4 hr CKD milling        |           | 2.85             | ≤ 20 nm       | 18,250                  | 114                         |

A comparison between the results governed by Alnahhal W. et al. and the present work related to the physical properties of CKD (sp. Gravity, surface area, initial time of setting) can be seen in figures 2, 3 and 4 respectively. The values of the specific gravity are little higher than that of Alnahhal W. et al [16], which means that removing alkali composites had improve the sp. gravity results.

Figure 2. Comparison between the results of CKD sp. gravity of Alnahhal W. and present work.
Figure 3 shows the relationship between the time of milling and the surface area in cm²/g and as it clearly seen from this graph that the present results gave upper bound results compared to Alnahhal W. et al. This difference in the results is surely due to the lack of alkali and the improvements in the whole surface area of CKD particles.

The reversed relation between the same variable (time of milling in hours) and the initial time of setting in minutes is shown in figure 4. All of the results are in the upper bound compared to Alnahhal W. et al [16]. except the final result which is in lower value.

3.2 Chemical properties of the un-milled and milled cement kiln dust.
The chemical properties of the milled and un-milled CDK are represented in table 5. It is clear from the table that the results of the milled CDK are absolutely near that of the un-milled samples of CDK and same conclusion was mentioned by Alnahhal W. et al [16]. The highest percentage ratios are lime and silica followed by sulfates, alumina and iron oxide. The composition of this milled CKD approximately includes a gradual decrease from the highest percentage ratios to the lowest exactly like that of the ordinary cement which means it’s using with cement as a fine or nano-material will be safe, approved and favorable.

Table 8. Chemical composition results of CKD after 4 hours milling.

| Compound     | Percentage ratios of compounds in dust before | Percentage ratio of compounds in dust after washing | Percentage ratio of compounds in CKD after 4 hours |
|--------------|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|
|              |                                               |                                               |                                               |
3.3. Compressive strength of the cement mortars

Figures 5, 6, 7 and 8 show the relationship between the compressive strength of mortars using an addition of CKD ratios 5%, 10%, 15% and 20% respectively. As it is well-known that as the curing time or age of mortar increases the compressive strength increases also. This fundamental note is clear through whole of these graphs but the new ensured truth is that all of the values of the compressive strength are increased in different ratios compared to the control mixture (un-milled CKD). The average increase can be shown in Table (9). From this table the ratios governed ensures that there is an increase in the compressive strength. Moreover, the ratios of increase for 7 days curing for whole ratios of CKD addition are high compared to other durations of curing. This means that an early high compressive strength is gained. That is due to the free lime presence in the CKD product.

3.3.1. Table of chemical analysis of CKD product

| Component     | Washing | Milling | Milling |
|---------------|---------|---------|---------|
| Silica SiO₂   | 15.46   | 16.02   | 16.45   |
| Alumina Al₂O₃ | 3.91    | 3.77    | 3.89    |
| Iron Oxide Fe₂O₃ | 3.05 | 3.49    | 3.67    |
| Lime CaO      | 43.40   | 44.45   | 44.88   |
| Magnesia MgO  | 2.98    | 3.03    | 3.26    |
| Sulfate SO₃  | 6.34    | 5.50    | 5.50    |
| Potassa K₂O   | 2.44    | 1.14    | 1.20    |
| Soda Na₂O     | 1.42    | 0.84    | 0.94    |
| Chloride Cl   | 0.29    | 0.247   | 0.247   |
| Loss on Ignition | 28.86 | 22.11   | 22.31   |
| L.O.I.        | 100.15  | 100.60  | 102.347 |
| Total         |         |         |         |
| Free lime     | 2.96    | 0.20    | 0.20    |

Figure 5. Results of compressive strength of mortar (5% CKD addition) and milling time during different curing times
Table 9. Percentage increase of mortar compressive strength with period of curing.

| CKD addition ratio | Average percentage increase in compressive strength compared to control mixture specimen |
|--------------------|----------------------------------------------------------------------------------------|
|                    | 7 days curing | 28 days curing | 56 days curing |

Figure 6. Results of compressive strength of mortar (10% CKD addition) and milling time during different curing times.

Figure 7. Results of compressive strength of mortar (15% CKD addition) and milling time during different curing times.

Figure 8. Results of compressive strength of mortar (20% CKD addition) and milling time during different curing times.
4. Conclusions and recommendations

The following conclusions are recorded as a result of the present work:

1. The authors ensure the conclusions of Alnahhal W. et al. through their work mentioned in the literature especially the increase in the compressive strength of the mortar mixes.

2. It was noted that the increase in the compressive strength in the present work is little higher than that of Alnahhal W. et al. So, this increment may be due to the lack in alkali which was washed from the CKD to some extent.

3. The initial setting time was started with a value higher than that of Alnahhal W. et al. and gradually decrease to reach a value less than that of Alnahhal W. et al. at the end. This variation may be due to the new composition of CKD after washing.

4. The washed alkali solution can be used in industries in so many ways.

5. The washed CKD can be returned to the cement manufacturing process instead of converting it to nano CKD if it is needed in an aim differs than the scope of this paper.

6. The results of the surface area of the milled CKD were increased as the time of milling increased and this truth ensures that fine and nano materials were gained and that will increase the compressive strength of mortar.

The following recommendations can be drawn here:

1. More precise tools and instruments were needed to reach better results like using High Energy Ball Mill Emax which has highly efficient liquid cooling cooled by an integrated water cooling system.

2. A future work might be planned and executed on concrete mixes or batches to see the effect of using CKD milled or un-milled on its properties.

3. Study the consistency of the concrete mixes when the CKD are added to it by certain ratios.

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