Draft Guidelines for Co-processing of Distillery spent wash concentrate in Cement Industry
1. Background

Co-processing of wastes in cement kiln is emerging as one of the environment friendly alternative disposal method in the recent years. Distillery spent wash has high organic content and dissolved inorganic solids which require careful handling and proper disposal to avoid damage to the environment. Presently it is being used on to land to provide nutrient value, though it contaminates both the ground water and surface water sources, as most often it is practiced in non-scientific manner. The adverse effect of spent wash disposal, via bio-composting is magnified during the rainy season. Concentration of spent wash in multiple effect evaporators and its subsequent incineration is also being practiced. However, it is not found technically and economically feasible in majority of cases. Hence, Central Pollution Control Board (CPCB) proposed, co-processing of concentrated spent wash in cement kiln.

There are around 400 distillery units in India, with a total production capacity of about 3800 million litres of alcohol. Average generation of spent wash is around 8-12 litres / litre of alcohol produced, depending on continuous or batch process and quality of molasses used etc.

Thus the co-processing of spent wash concentrate in cement kiln has following benefits:

(a) wastes are destroyed at a higher temperature of around 1200-1400 °C and longer residence time,

(b) inorganic content gets fixed with the clinker apart from using the energy content of the wastes leaving no residue behind

(c) the acidic gases, if any generated during co-processing gets neutralized, since the raw material in cement kiln is alkaline in nature, and

(d) such phenomenon also reduces resource requirement.

While in case of incineration, residuals require to be disposed suitably.

A typical physico-chemical characteristics of distillery spent wash is depicted at Annexure-I.

The production of cement in India is about 200 Million Tonnes per annum. The country, therefore, has potential to utilize spent wash concentrate from the distilleries for co-processing in cement industry.

Based on satisfactory performance of trial run, CPCB has granted regular permission for Co-processing of few categories of wastes and 25 cement
manufacturing units in various States have already started co-processing of permitted wastes. Further, trial studies are undertaken for various other wastes.

2. **Objective**

Despite of co-processing having inherent advantages, a careful & scientific study of trial run for spent wash with potential to damage environment during transportation, handling, storage and processing was done. The desired handling, transport and storage applicable to distillery spent wash have been described in the following sub-sections.

Distillery spent wash is not categorized as a hazardous waste, still co-processing of the same needs to be handled in an environmentally sound manner, avoiding the possibilities of environment contamination. These guidelines includes standard methodology for submission of application for streamlining the entire processing mechanism taking all essential safeguards along with the delivery of approvals in a reasonable time frame.

3. **Occupiers responsibility for handling of spent wash**

“Occupier” in relation to any factory or premises, means a person who has, control over the affairs of the factory or the premises and includes in relation to spent wash;

The occupier shall take all adequate steps while handling spent wash to:

(a) Contain contaminants and prevent leakages/spillages and limit their consequences on the environment; and

(b) Provide persons working on the site with the training, equipment and necessary information.

4. **Authorization/permission**

The concerned distillery shall obtain separate permission from SPCB/PCC for co-processing of spent wash concentrate in the prescribed formats. All other consents/authorization under the relevant acts shall also be valid for the industry.

The spent wash shall be collected, stored and co-processed only in the cement industries that have obtained necessary permission from the State Pollution Control Board/ Pollution Control Committee for the purpose in the prescribed format (Annexure II & III).

5. **Collection and transportation of Spent wash**

Safe transportation of spent wash to the site for co-processing in cement industry is a collective responsibility of the waste generator and processor as provided at Annexure - IV.
6. **Storage of Spent wash**

The occupier /co-processor/ cement industry may store the permitted quantity of spent wash concentrate for co-processing and shall maintain a record of transfer, storage and co-processing of such wastes and make these records available for inspection.

The detailed storage requirements for spent wash concentrate have been provided at Annexure IV.

7. **Feeding of spent wash concentrate for co-processing**

Different feed points can be used to inject the spent wash concentrate into the cement production process. The most common ones are:

- Main burner at the rotary kiln outlet end
- Pre-calciner

![A typical controlled injection system for spent wash parallel to that of pulverized coal, via the diesel firing line provided for start up of the kiln](image)

Appropriate feed points have to be selected according to the physical & chemical characteristics of the spent wash used.

8. **Suitability of spent wash for co-processing**
The findings of the trial runs at M/s Rajashree Cement Plant, Gulbarga, Karnataka and M/s Wadi Cement Works, Gulbarga, Karnataka suggest that disposal of spent wash concentrate through co-processing route is suitable and preferred way. The typical proximate analysis, ultimate analysis, chemical analysis and heavy metals and volatiles in spent wash are given at Annexure V. Co-processing of spent wash does not seem to influence clinker / cement quality or change kiln behavior subject to it when fed in limited and controlled low proportion of 3 - 5% of heat/coal substitution. Further trial runs may be attempted for co-processing of higher percentages.

9. Operating Conditions

Co-processing plants shall be designed, equipped, built and operated in such a way that the gas resulting from the co-processing is raised in a controlled and homogeneous fashion and even under the most un-favorable conditions, to a temperature of 950°C with a residence time of two seconds.

Co-processing plants shall operate in an automatic system to prevent spent wash feed, whenever emission monitoring show that any emission limits value is exceeded due to disturbances or failures of air pollution control devices.

Co-processing plants shall be designed, equipped, built and operated in such a way as to prevent emission into the air giving rise to significant ground level air pollution; in particular; exhaust gases shall be discharged in a controlled fashion by means of a stack, the height of which is calculated in such a way as to safeguard human health and the environment.

The management of the co-processing plant shall be in the hands of a skilled person, competent to manage the concentrated spent wash in an environmentally sound manner.

10. Air Pollution Control requirements

The dust emission is the main pollutant emanated from cement industry which is unlikely to change during co-processing of spent wash with moisture content. Generally cement kilns are equipped with Electro Static Precipitator or Bag House to control the particulate matter emission. Any acidic gases formed during co-processing are likely to be scrubbed by the raw material (lime stone) being of an alkaline nature and are incorporated into the cement clinker. However, cement industry has to ensure compliance of particulate matter emission standards during co-processing as prescribed in consent conditions issued by SPCB/PCC. For other pollutants i.e. HCl, SO₂, CO, TOC, NOx, Cd + Ti + their compounds, Hg and its compounds, Sb + As + Pb + Co + Cr + Zn + Cu + Mn + Ni + V + their compounds, the emission during co-processing should not exceed the base line emissions i.e.; during pre co-processing phase of trial run.

11. Emission standards
The emission standards for particulate matter prescribed for cement kiln by the concerned State Pollution Control Board shall be applicable during co-processing in cement kiln also. For other pollutants i.e. HCl, SO₂, CO, TOC, NOₓ, Cd + Tl + their compounds, Hg and its compounds, Sb + As + Pb + Co + Cr + + Zn + Cu + Mn + Ni + V + their compounds, the emission during co-processing should not exceed the base line emissions i.e.; during pre co-processing phase of trial run.

12. Monitoring requirements

The continuous measurement of particulate matter emission shall be carried out at co-processing plant and the emission data shall be submitted to SPCB/PCC as well as CPCB. As per directions of SPCB monitoring of other parameters will be done by the cement plant.

13. Application Procedure

a) Cement industries can submit their application in the desired format (Annexure VI) to SPCB/PCC with copy endorsed to CPCB for regular co-processing of distillery spent wash concentrate upto 3.5% of heat / coal substitution with the similar characteristics as given in Annexure V. SPCBs/PCCs shall grant permission for regular co-processing with copy endorsed to CPCB, within 45 days from the date of receipt of the application.

b) However, Cement Industries planning for the co-processing of spent wash concentrate at higher proportion, other than marked in para (a) shall obtain prior permission from SPCBs/PCCs. The procedure formulated for the same has been sequentially described in following subsections:

- The application duly filled in as per prescribed format for trail run (appended as Annexure VI) and enclosing all relevant enclosures has to be submitted to concerned State Pollution Control Board/PCC., where the spent wash is proposed to be co-processed., with a copy of complete application endorsed to CPCB by the proponent. In case CPCB has objections, if any, it shall communicate the same to the proponent with a copy to concerned SPCB/PCC within 30 days from the date of receipt of the application. The SPCB shall grant the permission for trail run within 60 days from the date of receipt of application.

- SPCBs/PCCs shall ensure that Cement industry planning to conduct trial run to co-process spent wash meets the emission standards prescribed by SPCB/PCC in consent order / environmental clearance.

- The protocol to be followed for trial run for co-processing has been detailed at Annexure VII. SPCBs/PCCs will grant permission for trial run in the format appended as Annexure II.
• The proponent shall inform SPCB & CPCB about the date of the trial run 15 days in advance so that SPCB & CPCB can supervise the trial run.

• After successful completion of trial run for higher percentage other than marked in para (a), for regular permission, the proponent shall apply to concerned SPCB/PCC in the prescribed proforma provided as Annexure VI along with all details related to the trial run with copy endorsed to CPCB. SPCB on receipt of the proposal will process the case. The proponent, if needed may be called for making a presentation.

• SPCB /PCC may grant or refuse the permission for regular co-processing within 30 days in format appended as Annexure III with copy endorsed to CPCB. The percentage of spent wash to be co-processed along with the characteristics shall be prescribed in the permission.

• Once regular permission for co-processing of distillery spent wash is granted, the other cement plants may not be required to conduct trial run. They can directly submit their application in the desired format to SPCB with copy endorsed to CPCB for regular co-processing. SPCB shall grant permission of regular co-processing with copy endorsed to CPCB, within 45 days from the date of receipt of the application.
**General Physico-Chemical characteristics of distillery spent wash**

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Characteristics</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Colour</td>
<td>Dark brown</td>
</tr>
<tr>
<td>2</td>
<td>Odour</td>
<td>Unpleasant burnt sugar</td>
</tr>
<tr>
<td>3</td>
<td>Specific gravity (g/cc)</td>
<td>1.2</td>
</tr>
<tr>
<td>4</td>
<td>pH</td>
<td>3.80</td>
</tr>
<tr>
<td>5</td>
<td>EC (dS/m)</td>
<td>43</td>
</tr>
<tr>
<td>6</td>
<td>TDS (mg/L)</td>
<td>91700</td>
</tr>
<tr>
<td>7</td>
<td>TSS (mg/L)</td>
<td>26560</td>
</tr>
<tr>
<td>8</td>
<td>TS (mg/L)</td>
<td>118260</td>
</tr>
<tr>
<td>9</td>
<td>BOD (mg/L)</td>
<td>43000</td>
</tr>
<tr>
<td>10</td>
<td>COD (mg/L)</td>
<td>128000</td>
</tr>
<tr>
<td>11</td>
<td>Organic Carbon (%)</td>
<td>3.7</td>
</tr>
<tr>
<td>12</td>
<td>Nitrogen (mg/L)</td>
<td>1460</td>
</tr>
<tr>
<td>13</td>
<td>Phosphorus (mg/L)</td>
<td>326</td>
</tr>
<tr>
<td>14</td>
<td>Potassium (mg/L)</td>
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<tr>
<td>15</td>
<td>Sodium (mg/L)</td>
<td>356</td>
</tr>
<tr>
<td>16</td>
<td>Calcium (mg/L)</td>
<td>6800</td>
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<tr>
<td>17</td>
<td>Magnesium (mg/L)</td>
<td>4384</td>
</tr>
<tr>
<td>18</td>
<td>Chloride (mg/L)</td>
<td>10650</td>
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<tr>
<td>19</td>
<td>Sulphate (mg/L)</td>
<td>3000</td>
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<tr>
<td>20</td>
<td>Copper (mg/L)</td>
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<tr>
<td>21</td>
<td>Manganese (mg/L)</td>
<td>9.2</td>
</tr>
<tr>
<td>22</td>
<td>Iron (mg/L)</td>
<td>24.6</td>
</tr>
<tr>
<td>23</td>
<td>Zinc (mg/L)</td>
<td>7.8</td>
</tr>
<tr>
<td>24</td>
<td>Carbonates (mg/L)</td>
<td>Nil</td>
</tr>
<tr>
<td>25</td>
<td>Bicarbonates (mg/L)</td>
<td>1530</td>
</tr>
</tbody>
</table>

Source: TNAU Coimbatore, TN
ANNEXURE - II

Model Permission for Trial run for co-processing of spent wash concentrate in cement kiln

Permission is hereby accorded for trial run for co-processing of spent wash concentrate from .......... in the cement kiln of M/s ............... subject to various conditions detailed below:

1. The generator of spent wash shall obtain prior permission from the State Pollution Control Board to conduct the trial run for co-processing of waste in cement plant.

2. The Cement plant shall accept spent wash only from those distilleries having permission to co-process in Cement kiln & valid consent under Air & Water Act from State Pollution Control Board.

3. The Cement plant shall estimate the quantity of spent wash required to conduct the trial run as per Annexure-6, Part A of this guideline. Only estimated quantity of spent wash shall be stored and co-processed in the plant during trial run.

4. Cement industry shall provide adequate covered storage space for spent wash and also ensure that there is no leaching of any pollutant. For transportation of proposed spent wash to conduct trial run for co-processing in cement kiln, guidelines of CPCB shall be followed.

5. Monitoring during the trial run shall be conducted as per the monitoring protocol (Annexure-6, Part A) of this guideline. Emission monitoring shall be conducted by recognized laboratory in consultation with SPCB/CPCB. Other tests related to quality of clinker & cement should be carried out and information as per Annexure-6, Part B, should be generated / collected.

6. The cement plant shall ensure the compliance of the conditions stipulated in the consents issued under the Water Act, 1974 and Air Act, 1981 during the trial run of co-processing of spent wash.

7. The cement plant shall comply with all the requirements in accordance with the Public Liability Insurance Act, 1991 as amended.

8. SPCB reserves the right to review / impose additional conditions or revoke, change or alter any of the terms and conditions.

Necessary arrangement shall be made to conduct the trial run smoothly. The final programme of the trial run shall be communicated to SPCB and CPCB within a month. The cement plant shall inform the date of trial run well in advance so that SPCB and CPCB representative can be present during the trial run. After trial run, the study report incorporating the information as at Annexure - 6 of the guidelines shall be submitted to SPCB for consideration.
Model Permission for co-processing of Spent Wash Concentrate in cement kiln

Permission is hereby accorded for co-processing of spent wash concentrate from ........... in the cement plant of M/s ................., subject to compliance of various conditions detailed following:

1. The permission is valid only for co-processing of spent wash concentrate. The waste characteristics should be similar to that for which trial runs have been conducted by SPCB / CPCB. Prior permission has to be obtained for co-processing of any other waste.

2. The generator of spent wash shall obtain permission from State Pollution Control Board to co-process spent wash concentrate in cement plant.

3. The cement plant shall estimate the quantity of spent wash required to be co-processed. Cement industry shall provide adequate covered space for storage of spent wash concentrate and also ensure that there is no leaching of any pollutant at any stage of processing. The actual quantity of spent wash co-processed in each calendar year shall be reported to the SPCB & CPCB.

4. The cement plant shall ensure the compliance of the conditions stipulated in the consents issued under the Air Act, 1981 and Water Act, 1974 during the co-processing of spent wash.

5. The emission standards for particulate matter prescribed for cement kiln by the concerned State Pollution Control Board shall be applicable during co-processing in cement kiln also. For other pollutants i.e.; CO, VOCs, TOC, NOx, HCl, F, SO$_2$, Cd + Ti + their compounds, Hg and its compounds, Sb + As + Pb + Co + Cr + Cu + Zn + Mn + Ni + V + their compounds, the emission values during co-processing shall not exceed the base line emissions i.e. during pre co-processing phase of trial run. The continuous measurement of particulate matter emission shall be carried out at co-processing plant and the emission data shall be submitted to the concerned SPCB/PCC & CPCB. As per direction of SPCB monitoring of other parameters will be done by the cement plant.

6. A log book of the waste co-processed shall be maintained including emission monitoring result during co-processing.

7. During co-processing of spent wash in cement kiln, the cement plant shall comply with all the requirements in accordance with the Public Liability Insurance Act, 1991 as amended.

8. In case of any violation in the conditions stipulated, the permission can be withdrawn at any time.
9. SPCB reserves the right to review / impose additional conditions or revoke, change or alter any of the terms and conditions.
Storage, Collection & Transportation of Distillery Spent Wash for Co-processing

1. **Storage Requirement**

1) The cement industry may store the spent wash concentrate up to seven days of permitted quantity for co-processing.
2) In order to have appropriate measures to prevent percolation of spills, leaks etc. to the soil and ground water, the storage area should be provided with concrete floor.
3) Measures should be taken to prevent entry of runoff into the storage area. The storage area shall be designed in such a way that the floor level is at least 150 mm above the maximum flood level.
4) The storage area floor should be provided with secondary containment such as proper slopes as well as collection pit so as to collect wash water and the leakages/spills etc.
5) All the storage yards should be provided with proper peripheral drainage system connected with the sump so as to collect any accidental spills in roads or within the storage yards.

2. **Storage Drums/Containers**

(a) The container shall be made up of MS steel.
(b) Drums containing wastes stored in the storage area should be labeled properly

3. **Spillage/leakage control measures**

(a) The storage areas should be inspected daily for detecting any signs of leaks or deterioration, if any. Leaking or deteriorated containers should be removed and ensured that the contents are transferred to a sound container.
(b) Proper slope with collection pits be provided in the storage area so as to collect the spills/leakages.

4. **Record Keeping and Maintenance**

Proper records with regard to the industry–wise waste received in the tanker and its characteristics need to be maintained.

5. **Miscellaneous**

(a) Good house keeping need to be maintained around the storage areas;
(b) To the extent possible, manual operations within storage area are to be avoided. In case of manual operation, proper precautions need to be taken, particularly during loading / unloading of spent wash in drums;
(c) A system for inspection of storage area to check the conditions of the containers, spillages, leakages etc. should be established and proper records should be maintained;
(d) Tanks /drums should be properly dyked and should be provided with adequate transfer systems;

6. **Transportation requirements**

The following are the requirements pertaining to the transportation of waste:
(a) Vehicle used for transportation shall be in accordance with the provisions under the Motor Vehicle Act, 1988, and rules made there under.
(b) Transporter shall possess requisite copies of the certificate (valid authorization obtained from the concerned SPCB/PCC for transportation of waste by the waste generator and operator of a facility) for transportation of waste.
(c) Vehicle should be fitted with mechanical handling equipment as may be required for safe handling and transportation of the wastes.
(d) Name of the facility operator or the transporter, as the case may be, shall be displayed.
(e) Vehicle shall be fitted with roll-on /roll-off covers if the individual containers do not possess the same.
(f) Each vehicle shall carry first-aid kit, spill control equipment and fire extinguisher.
(g) The driver of the transport vehicle shall have valid driving license from the State Road Transport Authority.
(h) Driver(s) shall be properly trained for handling the emergency situations and safety aspects. The design of the trucks shall be such that there is no spillage during transportation.

7. **Responsibilities of Transporter**

Transporter shall be responsible for:
(a) Obtaining requisite permission from SPCB/PCC or any other permission that may be required under the Motor Vehicle (Amendment) Act of 1981) for transportation of spent wash.
(b) The transport vehicles shall be designed suitably to handle and transport the spent wash concentrate.
(c) Transporting the wastes in closed container at all time.
(d) Delivering the wastes at designated points only.
(e) Informing SPCB/PCC or local authority, occupier / operator of a facility, and other concerned immediately in case of spillage, leakage or other accidents during transportation.
(f) Vehicle should be provided with facility to collect and Cleanup in case of contamination.
## TABLE 1: PROXIMATE ANALYSIS OF SPENT WASH CONCENTRATE

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Constituent</th>
<th>Value, %</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Moisture Content</td>
<td>47.01</td>
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<tr>
<td>2</td>
<td>Volatile Matter</td>
<td>35.69</td>
</tr>
<tr>
<td>3</td>
<td>Ash Content</td>
<td>10.59</td>
</tr>
<tr>
<td>4</td>
<td>Fixed Carbon</td>
<td>6.71</td>
</tr>
<tr>
<td>5</td>
<td>Calorific Value (kcal/kg)</td>
<td>2000</td>
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</table>

## TABLE 2: ULTIMATE ANALYSIS OF SPENT WASH CONCENTRATE

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Constituent</th>
<th>Value, %</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Carbon</td>
<td>19.92</td>
</tr>
<tr>
<td>2</td>
<td>Hydrogen</td>
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</tr>
<tr>
<td>3</td>
<td>Nitrogen</td>
<td>1.35</td>
</tr>
<tr>
<td>4</td>
<td>Sulphur</td>
<td>0.96</td>
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</table>
### TABLE 3: CHEMICAL ANALYSIS OF ASH OF SPENT WASH CONCENTRATE

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Percent (LOI free basis)</th>
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</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>1.23</td>
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<tr>
<td>Fe₂O₃</td>
<td>0.46</td>
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<tr>
<td>Al₂O₃</td>
<td>1.07</td>
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<tr>
<td>CaO</td>
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<tr>
<td>MgO</td>
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<tr>
<td>SO₃</td>
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<tr>
<td>Na₂O</td>
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<tr>
<td>K₂O</td>
<td>28.38</td>
</tr>
<tr>
<td>Cl</td>
<td>18.54</td>
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<tr>
<td>Item, %</td>
<td>Sample 1</td>
</tr>
<tr>
<td>--------</td>
<td>----------</td>
</tr>
<tr>
<td>SO₃</td>
<td>0.24</td>
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<tr>
<td>Cl</td>
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<td>Na₂O</td>
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<td>Moisture</td>
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<td>As</td>
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<td>Ba</td>
<td>0.007</td>
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<tr>
<td>Co</td>
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<tr>
<td>Cr</td>
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<td>Cu</td>
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<td>Mn</td>
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<td>Tl</td>
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</tr>
<tr>
<td>Zn</td>
<td>0.005</td>
</tr>
<tr>
<td>V</td>
<td>0.001</td>
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ANNEXURE - VI

Format* for obtaining approval from the State Pollution Control Board to utilise Spent wash in Cement Industries

<table>
<thead>
<tr>
<th>1</th>
<th>Name &amp; Address of the Unit:</th>
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</thead>
<tbody>
<tr>
<td>1.1</td>
<td>Contact Person and phone number:</td>
</tr>
<tr>
<td>1.2</td>
<td>Products to be manufactured and quantity (MTs/Day):</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>2</th>
<th>Details of source of spent wash to be utilized</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1</td>
<td>Name &amp; address of Distillery generating spent wash.</td>
</tr>
<tr>
<td>2.3</td>
<td>Generation (MTA) of spent wash proposed for utilization:</td>
</tr>
<tr>
<td>2.4</td>
<td>Detailed characteristics of spent wash (concentrated) proposed for utilization:</td>
</tr>
<tr>
<td>2.5</td>
<td>Process flow diagram of Distillery</td>
</tr>
<tr>
<td>2.6</td>
<td>Measures taken for storage &amp; transportation of concentrated spent wash</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>3</th>
<th>Details of utilization of Spent wash</th>
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</thead>
<tbody>
<tr>
<td>3.1</td>
<td>Process Flow Diagram indicating spent wash feeding mechanism</td>
</tr>
<tr>
<td>3.2</td>
<td>Please attach copy of air consent, water consent and authorization:</td>
</tr>
<tr>
<td>3.3</td>
<td>Base line data including characteristics pertaining to air emissions, wastewater generation and other solid wastes including hazardous waste being generated:</td>
</tr>
<tr>
<td>3.4</td>
<td>Material Balance without utilizing spent wash</td>
</tr>
<tr>
<td>3.5</td>
<td>Quantity of coal to be replaced by co-processing</td>
</tr>
<tr>
<td>3.6</td>
<td>Material balance with utilization of spent wash</td>
</tr>
<tr>
<td>3.7</td>
<td>Chemistry involved with and without utilization of spent wash:</td>
</tr>
<tr>
<td>3.8</td>
<td>Data including characteristics pertaining to air emissions, waste water generation and other solid wastes including hazardous waste being generated during utilization of spent wash, if available:</td>
</tr>
<tr>
<td>3.9</td>
<td>Details of findings of laboratory/ pilot scale study, international practice etc.</td>
</tr>
</tbody>
</table>

* To be filled and submitted by the cement unit, who desires to utilize distillery spent wash for co-processing.
ANNEXURE - VII

Protocol of Trial Run for Co-processing of Spent Wash in Cement Industry

Part – A

Requirement of testing of distillery spent wash

At least one representative sample shall be collected (for the whole trial period) and analysed for the following.

1) Calorific value of the waste (KCal / Kg) : (on wet and dry wt. basis)
2) Proximate analysis (Moisture, Ash, Volatile matter, Fixed carbon)
3) Ultimate analysis (Carbon content, Hydrogen content, Sulphur content, Nitrogen content, Oxygen content)
4) Characteristics of the waste
5) Total Organic Carbon (TOC)
6) Total Hydrocarbon
7) Organo - chlorine compounds
8) VOCs and Semi-VOCs
9) Poly Chloro Biphenyls (PCBs)
10) Poly Chloro Phenols (PCPs)
11) Viscosity
12) Moisture content
13) Solid content

Requirement of testing for conventional fuel

At least one representative sample shall be collected (for the whole trial period) and analysed for the following.

1) Calorific value of sludge (KCal / Kg) : (on wet and dry wt. basis)
2) Proximate analysis (Moisture, Ash, Volatile matter, Fixed carbon)
3) Ultimate analysis (Carbon content, Hydrogen content, Sulphur content, Nitrogen content, Oxygen content)
4) Characteristics of the fuel (Chlorine, Chloride, Fluorine, Potassium, Sodium, Sulphates and metal content (lead, tin, cadmium, arsenic, mercury, chromium, cobalt, nickel, thallium, copper, vanadium, antimony, manganese, selenium, lead)
5) Total Organic Carbon (TOC)
### Monitoring Programme at Cement Plant

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Date and duration</th>
<th>Operation of Cement Kiln</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>One Day</td>
<td>Emission monitoring during normal operation of cement kiln</td>
</tr>
<tr>
<td>2.</td>
<td>Three Days</td>
<td>Emission monitoring during trial run of cement kiln at a fixed percentage of spent wash feeding</td>
</tr>
<tr>
<td>3.</td>
<td>One Day</td>
<td>Emission monitoring during normal operation of cement kiln after stopping trial run</td>
</tr>
</tbody>
</table>

Detailed emission monitoring schedule to be followed before, during and after trial run of co-processing of distillery spent wash in cement kiln

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Parameter</th>
<th>Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Particulates</td>
<td>4 samples / day</td>
</tr>
<tr>
<td>2.</td>
<td>SO$_2$</td>
<td>4 samples / day</td>
</tr>
<tr>
<td>3.</td>
<td>HCl</td>
<td>4 samples / day</td>
</tr>
<tr>
<td>4.</td>
<td>CO</td>
<td>4 samples / day</td>
</tr>
<tr>
<td>5.</td>
<td>NOx</td>
<td>4 samples / day</td>
</tr>
<tr>
<td>6.</td>
<td>Total Organic Carbon</td>
<td>1 sample / day</td>
</tr>
<tr>
<td>7.</td>
<td>Fluoride</td>
<td>2 samples / day</td>
</tr>
<tr>
<td>8.</td>
<td>Hydrocarbons</td>
<td>2 samples / day</td>
</tr>
<tr>
<td>9.</td>
<td>VOC</td>
<td>2 samples / day</td>
</tr>
<tr>
<td>10.</td>
<td>PAH</td>
<td>2 samples / day</td>
</tr>
<tr>
<td>11.</td>
<td>Metals (both particulate and vapour phase)</td>
<td>1 sample / day</td>
</tr>
<tr>
<td></td>
<td>Cd, Th, Hg, Sb, As, Pb, Cr, Co, Cu, Mn, Ni, V, Zn, Sn, Se</td>
<td></td>
</tr>
<tr>
<td>12.</td>
<td>Dioxin &amp; Furan</td>
<td>1 sample / day</td>
</tr>
<tr>
<td>13.</td>
<td>Cyanide</td>
<td>1 sample / day</td>
</tr>
</tbody>
</table>
Ambient Air Quality Monitoring

AAQ monitoring shall be carried out at three locations (one in upwind and two in down wind direction).

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>PM$_{10}$</td>
</tr>
<tr>
<td>2.</td>
<td>SO$_2$</td>
</tr>
<tr>
<td>3.</td>
<td>NOx</td>
</tr>
</tbody>
</table>

The monitoring shall be carried out 24 hourly basis during whole trial period.

Clinker Analysis

Daily one representative sample shall be collected and analysed for the following during trial runs.

- Chlorine, Flourine, Sulphur, Cyanide, Chloride, Sodium
- Metals i.e. Cd, Th, Hg, Sb, As, Pb, Cr, Co, Cu, Mn, V, Zn, Sn, Se
- Leachability study of clinker (produced before trial run and during trial run) for fluoride, cyanide etc. including metals i.e. Cd, Th, Hg, Sb, As, Pb, Cr, Co, Cu, Mn, V, Zn, Sn, Se with proper conclusion
- Total Organic Carbon (TOC)

Raw Material Analysis

Atleast one representative sample shall be collected (for the whole trial period) and analysed for the following.

- Fluorides as F, Sulphates as SO$_4^{2-}$, Aluminium as Al$_2$O$_3$, Silica as SiO$_2$, Iron as Fe$_2$O$_3$
- Metals i.e. Cd, Th, Hg, Sb, As, Pb, Cr, Co, Cu, Mn, V, Zn, Sn, Se
- Total Organic Carbon (TOC)

Material Balance
A detailed report with complete interpretation including material balance for heavy metals and other important parameters such as Sodium, Chloride etc shall be prepared.

**Energy Balance**

A detailed report with complete interpretation on energy balance incorporating materials like coal, spent wash and if any others applicable shall be prepared.

**Part - B**

**Information required to be collected during the trial run by cement industry**

**General**

- Coloured computer print of process chart of different sections from Central Control Room
- Sketch showing stack duct connections and port hole with dimensions
- Note on spent wash handling and feeding mechanism i.e. from arrival of waste from generator to feeding in kiln burner / pyro
- Process flow diagram for co-processing of spent wash in cement kiln
- Distance between spent wash generator and cement plant.
- Copy of manual for transportation of spent wash and other related documents, if any
- Process flow diagram for cement manufacturing (Limestone stacking to packing plant)
- Layout plan
- Pyro drawings (with temp marking)
- Copy of consent order (showing prescribed standards)
- Stack emission and ambient air quality data of previous month
- Meteorology data (wind speed & direction, temp., rainfall) of trial period
- Wind rose diagram on daily basis
- Copy of daily log sheets of Kiln, clinker cooler and Coal mill for the entire period of trial run.
- Sketch showing location of ambient air quality stations w.r.t. cement plant (with marking of North, South direction)
- Photographs of trial run in CD
- Hourly data of continuous emission monitoring system for entire period of trial run (along with trend chart) for kiln section
- Note on manufacturing process of cement
- Kiln stoppages with date, time, duration and reason
- Coal mill stoppages with date, time, duration and reason
- Kiln ESP stoppages with date, time, duration and reason
Design (Optimum) Values

- Design raw mill output in TPH
- Design kiln feed in TPH
- Design clinker production in TPH
- Design coal consumption in kiln burner in TPH
- Design coal consumption in pyro in TPH
- Design temp. of pyro (max.)
- Design temp. of Kiln (burning zone)
- Average coal mill output in TPH in normal operation
- Average running hours of coal mill in normal operation
- Design coal mill output in TPH
- Gas residence time in kiln
- Gas residence time in pre-heater
Production data for entire trial period
- Raw mill output in TPH
- Kiln feed in TPH (with trend chart)
- Clinker production in TPH (Hourly average)
- Coal consumption in TPH in kiln (hourly average)
- Coal consumption in TPH in pyro (hourly average)
- Spent wash consumption in TPH (Hourly average)
- Temp. of pyroclone 6th stage maximum (hourly average)
- Temp. of kiln maximum at burning zone (hourly)
- Coal mill output in TPH (hourly)

Chemical analysis of clinker (hourly sample homogenized on daily basis) for entire trial period
- Loss on Ignition (LOI) %
- Silica (SiO2) %
- Iron Oxide (Fe2O3) %
- Aluminium Oxide (Al2O3) %
- Calcium Oxide (CaO) %
- Magnesium Oxide (MgO) %
- Sulphate (SO3) %
- Free Lime (CaO) %
- Total Alkalies
  a) Na2O %
  b) K2O %
- Minor Constituents
  a) P2O5 %
  b) Cl %
  c) Chloride

Physical tests of cement (hourly sample homogenized on daily basis) for entire trial period
- Blain (m2 / kg)
- Setting Time (minutes)
  a) Initial setting time
  b) Final setting time
- Soundness
  a) Le – Chat (mm)
  b) Autoclave (%)
- Compressive Strength (MPa)
  a) 3days
  b) 7days
  c) 28 days
Productivity Parameters during Trial Run
- Kiln Output Rate
  - (a) TPD
  - (b) TPH
- Energy Consumption
  - (a) Electrical, kWh/t Clinker
  - (b) Thermal, %
  - (c) Kcal/kg Clinker
  - (d) Coal Mill Power, kWh/t Coal

Kiln Parameters Observed During the Period of Trial
- Kiln Speed, rpm
- Kiln Torque, Amp.
- Kiln Feed, tph
- BE Temperature, °C
- BZ Temperature, °C
- PH Outlet (P-Line)
  - (a) Gas Temperature, °C
  - (b) Draft, mmWG
  - (c) O₂, %
  - (d) CO, %
- PH Outlet (K-Line)
  - (a) Gas Temperature, °C
  - (b) Draft, mmWG
  - (c) O₂, %
  - (d) CO, %
- Secondary Air temperature, °C
- Tertiary Air temperature, °C
- Moisture in Coal as fed, %
- Fine Coal residue, % on 90μ
- Litre Weight, g/l

Information related to Spent wash generator
- Address of the company with its background
- Name of the head of the company
- Year in which plant was commissioned
- List of the products manufactured with capacity and production data
- Manufacturing process of each product
- Process flow chart of each product (including waste generation)
- Waste water treatment plant details including capacity of tanks, retention time, characteristics of waste water, efficiency of ETP, sludge generation, process description of ETP, flow chart of ETP etc / Details of the process by which spent wash is generated
- Quantity of spent wash generation, present method of storage and disposal, cost of disposal
- Copy of air, water consent and authorization for waste disposal