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Development of a low-cost industrial waste treatment technology for resource conservation – An urban case study with gold-cyanide emanated from SMEs

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Abstract

Industries like electroplating, printed circuit board manufacturing and jewellery units situated in urban areas originate large-volume low-tenor effluents containing gold-cyanide. Since cyanide is toxic and gold being precious metal, non-renewable and finite resource; their complete removal from effluents is the key. The author in present paper, explores the possibility of treating gold-cyanide using an integrated biosorption-biodetoxification technologies. Rice husk, a low-cost biomass was selected from an exhaustive screening programme for the sorption of gold-cyanide. The optimum sorption conditions were: pH 4.0, biomass quantity 1% (w/v) and contact time 30-40 min. Uptake of gold-cyanide was found to increase as a function of initial gold-cyanide concentration. The data obtained obeyed the basic principles of Langmuir and Freundlich isotherm models. L-cysteine enhanced the biosorption ability of Rice husk. Adsorbed gold-cyanide on Rice husk could be desorbed using caustic soda with >90% efficiency. Traces of gold-cyanide remained after biosorption was then completely biodegraded using live bacterial consortium. The treated effluent conformed to statutory disposal standards in India.

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Keywords: Biosorption; Biodegradation, Gold-cyanide; Integrated system; Resource recovery; Rice husk waste biomass; SMEs

1. Introduction

Management of waste emanated by small and medium enterprises (SMEs) located in urban areas is one of the greatest challenges being faced by a developing and emerging economy like India. Industries

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like metal-plating, printed circuit board manufacturing and jewellery units originates large-volume low-tenor liquid effluents containing anionic metal-cyanides (MCNs) like gold-cyanide [Dicyanoaurate i.e. \([\text{Au(CN)}_2^-]\)] and silver-cyanide [dicyanoargentate i.e. \([\text{Ag(CN)}_2^-]\)] (Vapur et al. 2005). The total cyanide, gold and silver concentration in these effluents ranges from 5-25, 1-2 and 5-10 mg/l, respectively. In India, discharge limits for total cyanide is 0.2 mg/L (Santra, 2006), while for gold and silver the standards are not available. Since cyanide is toxic and gold & silver being precious metals, non-renewable and finite resource; their complete removal/recovery from effluents is the key. The most commonly adopted method for cyanide treatment is alkaline-chlorination (Ganczarczyk et al., 1985). Although this method is efficient in detoxifying free cyanide, it is not effective for MCNs (Eckenfelder, 1989). Other conventional physical-chemical methods are very expensive and not being used in India. At low concentrations, MCN recovery by conventional means is either not possible and/or very expensive. Biological methods like biodegradation using live microorganisms are subject to toxicity of cyanide and metals. Therefore, removal of precious resources like gold- and silver-cyanide from waste requires immediate attention of resource/environmental managers, scientists and technologists. The challenge is not limited only to their removal, but also extends to finding efficient and economical ways of possible recovery and recycling. It was thought that if an efficient management strategy and process for removal and recovery could be developed, precious MCN could be conserved, which in authors opinion would be a novel approach of resource recovery. Since dissociation of MCN in water forms anionic moieties (Sharpe, 1976), therefore, in principle, a well established physico-chemical methods can be used for removal and recovery of MCN. Overview on MCN removal mainly focuses on biodegradation (Patil & Paknikar, 2000) and biosorption of metal “cations” (Pethkar & Paknikar, 1998). Far less attention have been paid on biosorption of “anions” like gold-cyanide using low-cost biomass (Niu & Volesky, 2000) and has been an area of considerable research interest. Such process may provide an efficient, environment friendly and economical alternative to the conventional treatment processes. In light of above background, variety of low-cost biomass were used as sorbents to explore the possibility/feasibility of removing and recovering gold-cyanide using an integration of biosorption and biodegradation/biodetoxification process, the former being used for recovery of gold-cyanide and the latter for tackling unrecoverable gold-cyanide; and the case study of the same is presented.

2. Methodology

2.1. Biosorbents

Low-cost biomass used in the present study comprised of materials procurred from diverse sources in and around Kolhapur region and detailed category and type are mentioned in Table 1. All the samples were collected in polythene bags and transported to laboratory; washed several times with tap water followed by deionised water. Biomass samples were then dried at 50°C for 2-3 days and powdered using electric mixer and sieved to obtain particle size of \(\leq 500 \mu \text{m}\) (0.5 mm).

2.2. Gold-cyanide biosorption studies using low-cost waste biomass

A batch equilibration method was used to study sorption of gold-cyanide (0.02 mM i.e. 3.94 mg/l gold and 2.08 mg/l cyanide). Biosorbent (0.05 g) was contacted with 10 ml solution of gold-cyanide of desired pH (4.0) in a set of 50 ml capacity conical flasks. The flasks were incubated on rotary shaker incubator (150 rpm) at 30°C for 1 h. Contents of the flasks were filtered and analysed for residual gold and cyanide. All experiments were performed in duplicates and repeated twice to confirm the results. Appropriate controls were run simultaneously. In order to evaluate the influence of pH on biosorption of
Gold-cyanide, 10 g of powdered biomass was conditioned in pure water at pH 4, 5, 6, 7, 8, 9 and 10 prior to sorption. Gold-cyanide loading capacity (µmol gold-cyanide bound per gram weight of biomass) of selected biosorbent was determined by contacting 0.1 g powdered biomass several times with fresh batches of 10 ml gold-cyanide solution till saturation was achieved. For optimum biosorbent, gold-cyanide was contacted with varying amounts of biomass powder (0.1-5% w/v). Rate of gold-cyanide uptake was studied by contacting the biosorbent for a period ranging between 0-5 h. Under the optimised conditions, effect of various competing cations (0.01-0.1 mM) and anions (0.1-1 mM) on biosorption of gold-cyanide was checked. In order to test the effect of pre-treated biomass on uptake of gold-cyanide, the biosorbent were treated for one hour with various chemicals prior to sorption. Stock gold-cyanide was prepared as per Niu and Volesky (2000). Gold was analysed using Atomic Absorption Spectrophotometer (Elico, India). Total cyanide, chemical oxygen demand (COD), phosphates, sulphates and chlorides in solutions were determined as per Standard Methods (APHA-AWWA-WEF, 1998). pH and EC were measured using respective meters. pH of powdered biomass was determined as prescribed by Patil (2011).

2.3. Adsorption isotherm models

Gold-cyanide solutions of varying concentration (0.01 to 1 mM) was used to study the effect of initial gold-cyanide concentration on its adsorption. To obtain sorption data, gold-cyanide uptake value (Q) was calculated using the following equation:

\[ Q = V(C_i - C_f)/1000 \ m \] (1)

Where, Q is gold-cyanide uptake (µmol/g biomass); V is volume of solution (ml); C_i and C_f is initial and final concentration (µmol); m is mass of sorbent (g). The ‘Q’ value thus obtained was used to plot adsorption isotherms as per Freundlich and Langmuir equations (Freundlich, 1926; Langmuir, 1918):

\[ \ln Q = \ln K + (1/n) C_{eq} \quad \text{Freundlich equation} \] (2)

\[ C_{eq}/Q = (1/b Q_{max}) + (C_{eq}/Q_{max}) \quad \text{Langmuir equation} \] (3)

Where, C_{eq} is liquid phase concentration of gold-cyanide; b is Langmuir constant; Q_{max} is maximum gold-cyanide uptake; K is constant; n is number of metal reactive sites and Q is specific metal uptake.

2.4. Adsorption/desorption of gold-cyanide

Samples of 1 g biosorbent loaded with gold-cyanide was eluted using 1-5 N sodium hydroxide in concentrated form and analysed. Following elution of gold-cyanide, biosorbent was washed with pure water and was again conditioned to appropriate optimum pH to use in next adsorption/desorption cycle.

2.5. Biosorption of gold-cyanide from industrial wastewaters

Gold-cyanide containing wastewater sample was procured from gold plating unit. The effluent was characterized as per Standard Methods (APHA-AWWA-WEF, 1998). A batch equilibration method as mentioned earlier was employed. Rice husk (0.1 g) biomass (L-cysteine treated) was contacted with 10 ml (pH 4.0) of gold-cyanide effluent. Experiments were performed under optimum conditions. After contact, the contents were filtered and analysed for residual gold and cyanide.
2.6. Continuous biosorption studies using fixed bed column

Scale-up studies in continuous mode were conducted in fabricated fixed bed glass column for gold-cyanide biosorption. The column dimensions were: height 44 cm, internal diameter 1.3 cm, filter media height 30 cm, total volume 58.37 cm$^3$, working volume 39.80 cm$^3$. Column was filled with 21 g rice husk biomass pretreated with L-cysteine. Gold-cyanide effluent was passed through the bed column in upward direction (flow rate 40 ml/h) using programmable peristaltic pump (Enertech-Victor, India). All connecting tubes were of silicon. The effluent was passed upto 50 bed volumes till breakthrough (S-shaped curve) was obtained. Samples collected periodically were analysed for gold and cyanide.

2.7. Biodetoxification of unrecoverable gold-cyanide

Unrecoverable (residual) gold-cyanide in solutions after biosorption treatment was subjected to biodegradation using “live bacterial consortium” capable of degrading cyanide and thiocyanate (under aerobic conditions) previously isolated by enrichment culture technique (Patil, 2008). Optimum conditions for biodegradation were: pH 7.0, temperature 30°C, inoculum size 10$^7$ cells/ml and glucose 1 mM.

3. Results

3.1. Screening and selection of low-cost waste biomass for gold-cyanide sorption

Data in Table 1 summarizes the results obtained for gold-cyanide sorption under optimal pH conditions. Optimum sorption (Q) of gold-cyanide for all the biomass tested was at pH 4.0. Biosorption ceased above pH 7.0; while it increased in acidic pH. Other than activated charcoal, Rice husk (3.65 µmol/g) and Eichornia roots (3.56 µmol/g) were efficient biosorbents for gold-cyanide. Table 1 also shows the pH of all unconditioned biomass. pH of unconditioned Rice husk and Eichornia root biomass were 5.94 and 7.01, respectively, while their optimum pH of sorption after pre-conditioning was 4.0. On the basis of maximum gold-cyanide uptake obtained under optimum pH conditions, Rice husk and Eichornia root biomass were selected for further experiments. Activated charcoal was selected as a reference material.

3.2. Loading capacity of gold-cyanide

Table 2 depicts the data on gold-cyanide loading capacity of some selected pre-conditioned (at pH 4.0) biosorbents. Results were also compared with unconditioned biomass. It was found that Rice husk had maximum loading capacity for gold-cyanide (7.63 µmol/g) sorption followed by Eichornia root biomass (7.04 µmol/g). Loading capacity of activated charcoal was found relatively lower when compared with the Rice husk. Considering these results, selection of biosorbent was further narrowed down to Rice husk for gold-cyanide biosorption using conditioned biomass for further experiments.

3.3. Influence of biosorbent quantity and rate of gold-cyanide uptake

It could be seen from Fig. 1 that as the biomass quantity increased the % biosorption of gold-cyanide also increased. Maximum uptake in terms of Q (3.84 µmol/g) was observed at 1% (w/v) of Rice husk biomass. However, from 1 to 5% (w/v) there was no significant increase in biosorption. Data in Fig. 2 shows the rate of uptake of gold-cyanide by Rice husk biomass. The rate of uptake was maximum in first 15-20 minutes with over 80% of biosorption. Later, the sorption rate slowed down until it reached a
plateau after 30-40 min, indicating equilibration of the system. Maximum sorption of gold-cyanide (88.2%) was observed at 40 min.

Table 1: Biosorption of gold-cyanide at optimum pH

| Sr. No. | Biosorbent                        | pH of unconditioned biomass | Q (µmol gold-cyanide sorbed per gram biomass) |
|---------|-----------------------------------|----------------------------|-----------------------------------------------|
| (A)     | Agricultural waste/by-products     |                            |                                               |
|         | Coconut fibres                    | 4.24                       | 3.32 (4.0)*                                   |
|         | Cow dung cakes                    | 7.73                       | 3.07 (4.0)                                    |
|         | Groundnut shells                  | 5.49                       | 3.22 (4.0)                                    |
|         | Rice husk                         | 5.94                       | 3.65 (4.0)                                    |
|         | Rice straw                        | 6.13                       | 3.25 (4.0)                                    |
| (B)     | Industrial waste/by-products       |                            |                                               |
|         | Dairy waste sludge                | 6.88                       | 2.91 (4.0)                                    |
|         | Saw dust                          | 5.59                       | 3.52 (4.0)                                    |
|         | Sugarcane Bagasse                 | 5.92                       | 3.16 (4.0)                                    |
|         | Tea powder waste                  | 4.94                       | 2.94 (4.0)                                    |
| (C)     | Municipal solid waste components  |                            |                                               |
|         | Nirmalya (Waste flowers)          | 6.20                       | 3.43 (4.0)                                    |
|         | Compost                           | 7.28                       | 3.09 (4.0)                                    |
|         | Vegetable waste                   | 6.77                       | 2.91 (4.0)                                    |
| (D)     | Fungal and Bacterial waste/biomass |                            |                                               |
|         | Ganoderma sp.                     | 6.04                       | 3.01 (4.0)                                    |
|         | Yeast biomass                     | 4.39                       | 2.69 (4.0)                                    |
|         | Mucor heimalis                    | 4.45                       | 1.97 (4.0)                                    |
|         | Penicillium waste                 | 4.26                       | 3.08 (4.0)                                    |
|         | Streptomyces waste                | 4.86                       | 3.00 (4.0)                                    |
|         | Streptoverticillium waste         | 4.67                       | 2.77 (4.0)                                    |
|         | Wood rotting fungi                | 6.04                       | 3.18 (4.0)                                    |
|         | Bacterial consortium              | 6.83                       | 3.12 (4.0)                                    |
| (E)     | Algae biomass                      |                            |                                               |
|         | Mixed algal biomass               | 7.61                       | 3.29 (4.0)                                    |
| (F)     | Photosynthetic trees/plants waste |                            |                                               |
|         | Parthenium sp.                    | 6.69                       | 3.07 (4.0)                                    |
|         | Eichornia leaves                  | 5.57                       | 3.20 (4.0)                                    |
|         | Eichornia roots                   | 7.01                       | 3.56 (4.0)                                    |
|         | Eichornia stem                    | 5.58                       | 3.38 (4.0)                                    |
|         | Runners                           | 6.52                       | 3.06 (4.0)                                    |
|         | Tectona grandis leaves            | 5.40                       | 3.42 (4.0)                                    |
|         | Lantana camara leaves             | 6.59                       | 2.98 (4.0)                                    |
| (G)     | Reference materials               |                            |                                               |
|         | Activated charcoal                | 5.59                       | 3.80 (4.0)                                    |
|         | Bagasse Fly ash                   | 8.75                       | 3.40 (4.0)                                    |
|         | Control (without biomass)         |                            | 0.20 (4.0)                                    |

All values presented in table are average of two readings; *Values in parentheses indicates optimum pH

3.4. Adsorption isotherm models

Equilibrium sorption capacity of the sorbent increased with increasing initial concentration of gold-cyanide due to increase in the number of ions competing for the available binding sites in the biomass. The uptake of gold-cyanide approached towards plateau above 0.5 mM. To examine the relationship between sorption, isotherm models are widely employed for fitting the data. Data obtained showed that gold-cyanide uptake could be well fitted to Langmuir and Freundlich isotherm models with regression value > 0.98.
Table 2. Gold-cyanide loading capacity of selected biosorbents

| Biosorbent         | Loading capacity (µmol/g of biomass) | Remarks          |
|--------------------|--------------------------------------|------------------|
|                    | Conditioned biomass (at optimal pH) | Unconditioned biomass (at original pH of biomass) |
| Rice husk          | 7.63 (4.0)                           | 7.05 (5.94)      | Moderately affected |
| *Eichornia* roots  | 7.04 (4.0)                           | 4.38 (7.01)      | Significantly affected |
| Activated charcoal | 7.61 (4.0)                           | 7.58 (5.59)      | Not affected         |

All the values presented in table are average of two readings.

3.5. Influence of cations, anions and pre-treatment on gold-cyanide biosorption

Biosorption process was affected in the presence of zinc, chromium and cadmium by 33-40%, while it was consistently >80% in the presence of other metal cations/anions. Rice husk treated with 1% L-cysteine enhanced the biosorption capacity of gold-cyanide. It was therefore thought to determine the loading capacity of Rice husk pre-treated with L-cysteine. It was found that loading capacity of Rice husk biomass enhanced the biosorption of gold-cyanide up to 175% compared to untreated biomass.

3.6. Adsorption/desorption of gold-cyanide

The loaded gold-cyanide on Rice husk biomass could be desorbed with > 97% efficiency using 1 N sodium hydroxide solution. Final concentration in the eluent was 28-30 folds of initial concentration of gold-cyanide. However, during second cycle of biosorption, the loading capacity decreased by 10-15%.

3.7. Biosorption of gold-cyanide from industrial wastewaters

Table 3 depicts the data on gold-cyanide before and after biosorption. Gold and cyanide removal efficiency from gold-cyanide effluent was 91.53% and 82.69%, respectively. The residual cyanide in solution after biosorption was 0.59 mg/l, and unable to meet the statutory limits (0.2 mg/l) prescribed in...
India. Overall, the results indicated that Rice husk biomass (pre-treated with L-cysteine) was very effective in treating industrial effluent by biosorption process.

Table 3. Biosorption of gold-cyanide from industrial effluent in batch mode using rice husk pre-treated with L-cysteine

| Physicochemical parameters | Before biosorption | After biosorption | % Removal efficiency |
|----------------------------|--------------------|-------------------|----------------------|
| Colour                     | Colourless         | Colourless        | -                    |
| Turbidity                  | Clear              | Clear             | -                    |
| pH                         | 6.87               | 4.12              | -                    |
| Total cyanide              | 3.41               | 0.59              | 82.69                |
| Gold                       | 1.30               | 0.11              | 91.53                |
| Silver                     | 0.48               | 0.03              | 93.75                |
| Copper                     | 0.95               | 0.18              | 81.05                |
| Zinc                       | 0.50               | 0.10              | 80.00                |
| Iron                       | 0.11               | BDL               | 100.00               |
| Phosphates                 | 97.9               | 76.1              | 22.26                |
| Sulphates                  | 63.5               | 61.3              | 3.46                 |
| Chlorides                  | 173.0              | 155.2             | 10.28                |
| Chemical oxygen demand (COD)| 42                 | 31                | 26.19                |

All the figures given in the table are in mg/l, except pH; BDL: Below detectable limits

3.8. Biodegradation of unrecoverable (residual) gold-cyanide

Residual concentration of gold and cyanide in gold-cyanide effluent after biosorption treatment were 0.11 and 0.59 mg/l, respectively. The solution when subjected to biodegradation under optimum conditions, the consortium culture could degrade the cyanide from the effluent with an efficiency > 90% within 5 h. pH, cyanide and COD of the treated effluent were within the statutory limits (Table 4). Gold was not detected in bacterial free treated solutions. Findings indicated that biodegradation could be used as a polishing step in the treatment of gold-cyanide containing wastewaters.

Table 4. Treatment of residual gold-cyanide by a cyanide and thiocyanate degrading heterotrophic bacterial consortium

| Parameter                     | Biodegradation | *BIS Standards | % Removal Efficiency |
|-------------------------------|----------------|-----------------|----------------------|
|                               | Before         | After           |                     |
| pH                            | 6.99-7.04      | 7.05-7.12       | 5.5 – 9.0            |
| Total cyanide                 | 0.59 mg/l      | 0.04            | 0.2                 | 93.22%               |
| Chemical oxygen demand (COD)  | 102 mg/l       | 23 mg/l         | 250                 | 77.45%               |
| Gold                          | 0.11           | BDL             | Not Available        |

*BIS- Bureau of Indian Standards; BDL-Below Detectable Limits; All the values presented in table are average of two readings

3.9. Biosorption of gold-cyanide effluent in packed bed column and costing of process

Fig. 3 shows the breakthrough curve obtained for gold-cyanide biosorption. The breakthrough point (service time) observed was 60 h for both gold and cyanide. Total volume of effluent passed through the column was equivalent to 50 bed volumes. Biosorbent column got saturated after 90 h. For the present
The main objective of the present work was to study the feasibility of removal and recovery of gold-cyanide from industrial effluents using low-cost waste biomass followed by biodegradation. It is known waste biomass have high metal-binding capacity due to their inherent chemical nature, which is essentially required for application of biosorption process (Voilesky, 2003). Biosorption process is influenced by solution pH (Paknikar et al 2003). It was observed that maximum biosorption of gold-cyanide took place at acidic pH (pH 4.0). Rice husk and Eichornia root biomass were efficient biosorbents for gold-cyanide. Increased biosorption under acidic conditions might be due to the protonation of biosorbent surface (i.e. functional groups) acquiring net positive charges. Therefore,
probably the formation of species such as H+-AuCN₂⁻ on the biomass might have taken place. Thus, relatively more gold-cyanide species could have accommodated on the biosorbent sites. Since gold-cyanide being a stable complex, possibility of formation of hydrogen cyanide (HCN) gas is ruled out under acidic conditions (Sharpe, 1976). MCN containing industrial effluents have pH range of 6.0-10.0. Obviously, appropriate pH adjustments of the effluents will have to be carried out prior to biosorption. Loading capacity of biosorbent could be taken as an equivalent measure of binding sites present. It was observed that loading capacity of Rice husk was higher than Activated charcoal. This opens up new possibilities of developing an efficient biosorption technology for the recovery of anionic species like gold-cyanide and similar other contaminants.

For cost-effective treatment of industrial waste, it is critical to know the biomass quantity required. Maximum uptake was observed at 3% (w/v) of Rice husk biomass. This could be attributed to the interference between binding sites at higher quantities (deRome & Gadd, 1987). Process of biosorption is essentially a surface interaction and is characterized by rapid uptake (kinetics) of ions by biomass surfaces. Rapidity of process makes it a good candidate for use in effluent management on a large scale. In our studies, gold-cyanide uptake by Rice husk was rapid. Rapid kinetics has significant practical importance as it will facilitate smaller reactor/column volumes ensuring efficiency and economy. Effect of initial gold-cyanide concentration on biosorption by Rice husk showed that equilibrium sorption capacity of the sorbent increased with increasing initial concentration of gold-cyanide. This was due to the increase in number of ions competing for the available binding sites on biomass. Uptake of gold-cyanide at various concentrations reached a plateau at 0.5 mM (500 µmol). This may be attributed to the saturation of binding sites, which clearly showed that gold-cyanide uptake by Rice husk biomass was a chemically equilibrated and saturable phenomenon. Despite complexity of adsorption process, which can include several mechanisms, adsorption isotherms have been used to characterize MCN uptake and they appear to be of use for projected industrial applications (Tsezos & Volesky, 1981). Hence, it was decided to fit the available gold-cyanide sorption data with two most widely accepted adsorption models, viz. Freundlich and Langmuir. Linear transformation of the adsorption data using Freundlich and Langmuir models (R² = >0.98) allowed computation of gold-cyanide adsorption capacities. Experimental data obtained obeyed basic principles underlying these models. Enhancement of gold-cyanide biosorption by 175% using L-cysteine treated Rice husk in our studies supports the results of Niu and Volesky (2000). The anionic species [Au(CN)₂⁻] adsorbed by ionisable functional groups on cysteine loaded biomass carrying a positive charge when protonated. Recovery of target species from effluents is desirable when the species is costly and rare. Although concentration of such species in effluents may not be very high (<1-10 mg/l), but the total amount of target species may be quite significant considering the large volumes of effluent generated daily. For an effective and viable biosorption technology, elution methods for the recovery of target species should be highly efficient, economical and should not cause damage to the biomass. Sodium hydroxide was such eluting agent, capable of desorbing the loaded gold-cyanide on Rice husk. Final concentration of gold-cyanide in the eluent was 28-30 folds of the initial concentration. Such concentrated solution of recovered gold-cyanide may be recycled in the user industry. Experiments on industrial effluent in batch mode and continuous mode clearly showed that Rice husk biomass effectively removed gold-cyanide from the effluents.

Thus, it could be concluded that Rice husk waste biomass has immense potentials “as biosorbent” for the management of low tenor precious pollutants, as evident from the example of gold-cyanide. In the recent times, there has been a paradigm shift in the waste management practices, which is rapidly changing its face and orientation. Waste being regarded as a ‘resource’, could potentially be recovered from the effluents using suitable technologies. Therefore, in a real sense, concepts like recycling/recovery of waste resource are gaining remarkable importance in Anthrosphere. Applications of concepts similar to this work will ultimately reduce the demand for natural resources thereby prolonging its sustenance. In
this view of this the present work adds to the advancement of knowledge in the field of resource and pollution management, waste minimization and will help profitability of business community at large.

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