



Utilization of concentrate of membrane filtration of bleach plant effluent in brick production

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ABSTRACT

Utilization potential of membrane filtration retentate (concentrate) of bleach plant effluent from paper industry, in bricks production, was investigated in the present study. Bricks were prepared by using retentate of membrane filtration in place of water. The physical properties measured for bricks were dimensions, density, moisture content, compressive strength, water absorption and porosity. Chemical analysis of the bricks was performed for water leachability and acid leachability, using ASTM Standard methods D 3987-06 and D 5233-92 respectively. The leachate samples were analyzed for Al, Cu, Fe, Co, Cr, Pb, Ni and Zn using AAS (atomic absorption spectrophotometer). Adsorbable organic halides (AOX) of leachate were analyzed by using AOX analyzer model ECS 1200 using column method. It was observed that the physical and chemical properties of bricks especially fire clay bricks were not adversely affected by the use of wastewater. Therefore, the organic matter of the retentate of membrane filtration creates no problems for this application.

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1. Introduction

Disposal of concentrate (retentate) obtained from membrane filtration (10–25% of feed) of bleach plant effluent from paper industry, is a major problem. Many attempts have been made for treatment/disposal of concentrate using conventional biological treatment or land filling. Concentrate obtained from membrane treatment of bleach plant effluent of pulp mill has high pollutants in terms of COD (chemical oxygen demand), TDS (total dissolved solids) and AOX (adsorbable organic halides). If this concentrate is directly sent to the ETP (effluent treatment plant) for treatment, concentration of pollutants will increase. By this direct mixing, the volume of the wastewater from the bleach plant is reduced but the pollution load in terms of concentration is increased.

The by-products and residues from the pulp and paper industry are managed by using several approaches including aerobic and anaerobic treatment at ETP, land filling, incineration, in cement plant, brick-kilns, agricultural use and composting, recycling after advance treatments [1–3]. Organic residues are extensively used as pore formers in brick-kilns. Paper making sludge or residue is one of the best known organic pore-forming materials. It contains both organic and inorganic materials such as lime, kaolin or bentonite,

etc. [4]. Sutcu and Akkurt [5] have used recycled paper processing residues in making porous bricks with reduced thermal conductivity. Demir et al. [6] used kraft pulp production residues in clay brick production. Though most of the published work is mainly focused on the utilization of solid waste for bricks production, yet some studies have also been made for the use of wastewater for brick production. The use of three-phase olive mill wastewater in the manufacture of fired clay bricks has also been reported [7–9].

Brick is one of the important components for building construction industry. If every Indian is to be provided a roof above his/her head, the planning commission has estimated that India needs about 77 millions houses in urban areas and 63 million in rural areas by 2021 [10]. In India, different practices for the manufacture of handmade bricks are adopted in different regions of the country. In the brick industry, all operations, such as preparation of clay, molding, drying, and firing are carried out in open. The kiln, most commonly used in India is the Bull's trench kiln, but in majority of the cases open kiln is used in interior areas. Presently bricks are made from a mixture of plastic clays, and other additives like Fly ash, sandy loam, rice husk ash, ballast stone dust, etc. [11].

In view of the huge demand, of the order of 100 billion building bricks per annum, along with non-availability of suitable soil and depleting resources, the need to explore alternative raw materials and energy efficient technologies for making bricks has been realized [12]. Serious efforts have been made by many researchers for making bricks using different types of industrial wastes in the recent past such as Fly ash, blast furnace slag, etc. [4,13–15].

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Table 1
Characteristics of combined retentate.

Retentate	pH	Total Solids (mg/l)	COD (mg/l)	Color (Pt-Co U)	AOX (mg/l)
Extraction stage	8–10	11560–13886	5400–7348	1238–1628	54–68
Combined bleach plant ^a	7–8	9180–11890	4000–5906	1067–1310	72–98

Characteristics of combined retentate of UF, NF and RO (equal quantities of UF, NF and RO retentate were mixed) membranes of E-stage and combined bleach plant.

^a The combined bleached plant effluent consists of chlorination, extraction, and hypochlorite stage effluent.

The fly ash is a by-product of coal based thermal power plants and its production has already reached a staggering figure of almost 175 million tons per year. Such huge generation of ash from the thermal power station has already become alarming due to severe constraints of available land for dumping. For disposal of huge amount of fly ash generated (175 million tons per year by the end of 2007) 1,000,000 acres of valuable land is required for the construction of ash storage ponds. As per the estimation, utilization of fly ash can be targeted at least to an extent of 40–50% in different fields. The bricks needed for house construction, in urban and rural areas, shall consume 160 million tons of top soil, making barren 30.50 hectares of fertile land and shall need 19.2–20.8 million tons water.

In India, today there are about 600 pulp and paper mills with an annual production of 9.2 million tons of paper. Total effluent generation from bleaching section of a typical Indian paper mill ranges from 17 to 22 m³/ton of paper production. In this extraction stage contributes around 12–16 m³/ton of paper production. Industry is discharging huge amount of wastewater, while raw water which is being used in brick making can be used for drinking purpose. Time has come for the industry to plan for zero discharge. The membrane treatment reduces the effluent volume but does not lead to zero discharge. So in literature, the studies on the zero discharge of bleach plant effluent is not yet available, nor the utilization of pulp and paper mill wastewater for brick making is available.

The main objective of this study is to investigate the utilization potential of “membrane filtration retentate” in bricks production in place of water. Pulp residue can be effectively used for pore forming up to 5% additional level [6].

In the present study, two types of bricks were prepared using membrane filtration retentate of bleach plant effluent in place of water.

- Cement–fly ash bricks.
- Fired clay bricks.

Table 2
Chemical analysis of cement, fly ash and yellow clay (% by weight).

Chemical constituents	Cement	Fly ash	Clay
SiO ₂	18.6	30	65.9
Al ₂ O ₃	6.4	20	15.0
Fe ₂ O ₃	3.0	15	10.2
CaO	61.2	24	0.0
MgO	2.9	3	3.8
SO ₃	4.3	3	–
Na ₂ O	0.4	2	0.8
K ₂ O	1.0	1	1.1
Na ₂ O ^a	1.0	–	–
TiO ₂	0.2	–	–
P ₂ O ₅	0.1	–	–
MnO	–	–	0.2
LOI	0.9	2	3.0

Method of analysis: energy dispersive X-ray spectroscopy analysis; using EDS link ISIS 300 System and Scanning Electron Microscope, JSM-5600.

^a Acid-soluble alkali, LOI = loss-on-ignition (at 550 °C).

2. Materials and methods

2.1. Raw materials used in the bricks production

2.1.1. Stabilized cement–fly ash bricks (SCFBs)

The main constituent materials used in the production of cement–fly ash bricks are

- The L&T Ultra Tech, 53 grade ordinary Portland cement, batch no. 02 (week) 05 (month) 2008 (year), conforming to IS: 12269-1987 was procured in bulk for experiments.
- Fly ash of thermal power plant.
- Retentate of membrane filtration in place of water (retentate of two kinds of effluent were used one extraction bleach effluent and other combined bleach plant effluent. Characteristics of retentates are given in Table 1.

2.1.2. Fired clay bricks (FCBs)

In fired clay bricks, the following materials were used

- Yellow clay.

Table 3
Physical properties of raw materials.

Properties	Cement	Clay	Fly ash
Field dry density	–	1.900 g/cm ³	–
Field Moisture	–	9.40%	–
Specific gravity	3.15	2.644	–
Bulk density	1.5 g/cm ³	1.534 g/cm ³	0.98 g/cm ³
Voids	52.10%	1.534 g/cm ³	–
pH	12.5	8.5	–
Particle size distribution			
D10	2.76 μm	2.09 μm	–
D30	9.18 μm	14.38 μm	–
D60	22.10 μm	52.43 μm	–
D90	53.32 μm	106.02 μm	–
Coefficient of curvature, CC	1.38	1.88	–
Coefficient of uniformity, Cu	8.01	25.03	–
Fineness	245 m ² /kg	–	–
Mineral phases	Hartruite	Quartz	–
	Larinite	Anorthite	–
	Periclase	Chabazite	–
	Chabazite	Ca, Mg, Al silicate	–
	Grossular ferrian	–	–
Plasticity Index	–	28.03	–
Setting Time	–	–	–
Initial	115 min	–	–
Final	375 min	–	–
Soundness (by lechatelier)	0.5 mm	–	–
Compressive strength			
3 days	296 kg/cm ²	–	–
7 days	398 kg/cm ²	–	–
28 days	550 kg/cm ²	–	–
Water holding capacity (%)	–	–	66.8
Cation exchange capacity (%)	–	–	3.52
Sand (%)	–	–	65.9
Clay (%)	–	–	10.5
Silt (%)	–	–	1
pH (1:2)	–	–	6.5
pH (1:1)	–	–	6.11
ECH ₂ O (1:2) (dS/m)	–	–	0.33
ECH ₂ O (1:1)	–	–	0.23

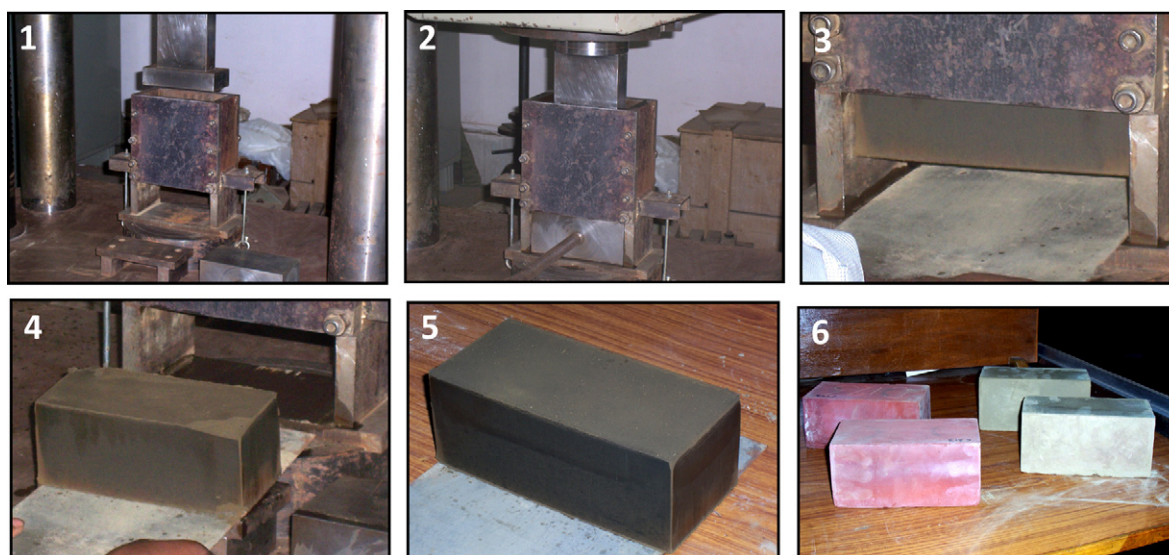


Fig. 1. Different stages in the manufacturing of SCFB and FCB. Stages: (1) installation of the die, (2) compression of the matrix, (3) ejection of the bricks, (4), removal of the bricks, (5) green bricks (6) cured SCFB and fired FCB.

- Retentate of membrane filtration in place of water (as given in Table 1).

Chemical and physical properties of cement, fly ash, and yellow clay are presented in Tables 2 and 3, respectively.

2.2. Bricks preparation

2.2.1. Stabilized cement fly ash bricks (SCFB)

Three combinations of cement + fly ash were prepared (i) 15% cement + fly ash, (ii) 30% cement + fly ash and (iii) 45% cement + fly ash. All the three combinations were mixed with 13% retentate by weight of OD (oven dry) raw material (two kinds of retentate of membrane filtration of E-stage effluent (EF) and combined bleach plant effluent (KF) separately with each combination). Raw materials were blended manually first in the dry state in the above-mentioned proportions and then the retentate (water) was mixed thoroughly.

Each brick was made with weight of 500 g material consisting of cement and fly ash in the above three combinations. Brick mixtures were compressed on the universal testing machine with 150 kg/cm² molding pressure [14]. Dimensions of bricks are 100.5 mm × 50.7 mm × 47.6 mm for SCFB (average of 50 samples).

The stabilized bricks were cured in two stages, humid curing followed by water curing. In humid curing, the bricks are kept in humid or moist state by sprinkling water on them for at least twice a day for a period of 2 days. The process not only helps in preventing cracking of bricks owing to uneven drying, but also gives maximum

strength to the bricks. During the curing process, the bricks were kept under shade, covered with jute sheets to protect them against direct sunlight. Bricks were submerged in water for the next 26 days, to get full strength after curing [16].

2.2.2. Fired clay bricks (FCBs)

In the fired clay bricks, clay was taken as raw material and mixed with 12% retentate by weight of OD raw material (two kinds of retentate of membrane filtration of E-stage effluent (EF) and combined bleach plant effluent (KF) was used separately). Each brick was made with 500 g clay. Bricks' mixture was compressed on the universal testing machine with 150 kg/cm² molding pressure [14]. Bricks were dried to approximately 5–7% moisture content (in 7 days). After drying, bricks were fired in the furnace at 925 °C temperature for 1 h [14]. Dimensions of bricks are 94.8 mm × 47.6 mm × 41.5 mm for FCB (average of 20 samples).

The different stages in the manufacture of SCFBs and FCBs are presented in Fig. 1. The stages involve (1) installation of the die, (2) compression of the matrix, (3) ejection of the bricks, (4) removal of the bricks, (5) green bricks and (6) cured SCFBs and fired FCBs [14].

2.3. Assessment of physical and chemical properties of SCFBs and FCBs

Using the relevant procedures code, the physical properties were measured for SCFBs and FCBs. The estimated parameters were physical dimensions, density and moisture content [17], compressive strength [18], water absorption and porosity [19].

Table 4
Physical properties of SCFB and FCB bricks.

Sample	Moisture content (%)	Bulk density (g/cm ³)	Dry density (g/cm ³)	Water absorption (%)	Porosity fraction	Compressive strength (kg/cm ²)
EF 15	23.36	1.70	1.38	30.62	0.51	127.03
EF 30	21.07	1.73	1.43	26.78	0.46	157.94
EF 45	11.97	1.87	1.67	13.61	0.25	281.15
KF 15	24.06	1.71	1.38	31.70	0.54	118.03
KF 30	17.38	1.81	1.54	21.24	0.38	275.01
KF 45	13.20	1.85	1.64	15.55	0.28	269.03
EC	9.44	2.11	1.92	10.43	0.22	457.12
KC	9.60	2.11	1.93	10.63	0.22	420.03

Abbreviations: EF = cement fly ash bricks with membrane filtrate concentrate of extraction stage effluent; KF = cement fly ash bricks with membrane filtrate concentrate of combined bleach plant effluent; EC = fired clay bricks with membrane filtrate concentrate of extraction stage effluent; KC = fired clay bricks with membrane filtrate concentrate of combined bleach plant effluent; 15, 30 and 45 in samples are the percentages of cement in that particular brick.

Table 5
Characterization of leachate of acid leachability test of bricks.

Sample	Al (ppm)	Cu (ppm)	Fe (ppm)	Co (ppm)	Cr (ppm)	Ni (ppm)	Pb (ppm)	Zn (ppm)	AOX (ppm)
KF15	1.084	0.051	0.1905	0.1905	0.138	0.0715	0.18	0.1435	5.98
KF30	0.352	0.0515	0.1755	0.248	0.083	0.158	0.3025	0.097	1.66
KF45	0.4375	0.0645	0.2265	0.1415	ND	0.1975	ND	0.1395	2.88
EF15	0.687	0.046	0.1495	0.07	ND	0.333	ND	0.1355	3.06
EF30	ND	0.04	0.1925	0.099	ND	0.3375	ND	0.1775	2.18
EF45	ND	0.0435	0.16	0.0525	ND	0.283	ND	0.1815	2.8
EC	0.673	0.0465	0.3355	ND	ND	0.227	ND	0.3405	0.79
KC	0.383	0.035	0.4025	ND	ND	0.1845	ND	0.2705	0.84
WF45	0.540	0.041	0.182	0.125	ND	0.220	ND	0.1152	ND
WC	0.228	0.038	0.317	ND	ND	0.185	ND	0.2853	ND
US EPA limit					1.1		0.5	0.535	

Abbreviations: EF = cement fly ash bricks with membrane filtrate concentrate of extraction stage effluent; KF = cement fly ash bricks with membrane filtrate concentrate of combined bleach plant effluent; EC = fired clay bricks with membrane filtrate concentrate of extraction stage effluent; KC = fired clay bricks with membrane filtrate concentrate of combined bleach plant effluent; WF = fly ash brick with natural water; WC = clay brick with natural water; 15, 30 and 45 in sample are the percentages of cement in that particular brick.

The chemical analysis of the SCFBs as well as FCBs was done for water leachability and acid leachability using ASTM Standards methods [20,21], respectively. The leachate samples were analyzed for heavy metals i.e. Al, Cu, Fe, Co, Cr, Pb, Ni and Zn using AAS (atomic absorption spectrophotometer). Adsorbable organic halides (AOX) of leachate were analyzed by using AOX analyzer model ECS 1200 using column method.

3. Results and discussion

Physical and chemical properties of the bricks are shown in Tables 4–6. Result of each physical property is an average of seven samples, except compressive strength. For compressive strength, results are average of three samples. In chemical analysis, results are average of two samples.

3.1. Moisture content and porosity fraction

The results of the moisture content and porosity fraction of samples with different proportions of cement–fly ash and fired clay bricks are shown in Table 4. Average moisture content in SCFBs vary between 11.97 and 24.06%. Higher moisture content was observed in the bricks made with low percentage of cement. In the FCBs, moisture contents were found to be between 9.44 and 9.60%. FCBs show less moisture content than SCFBs. As far as porosity fraction is concerned, it varies from 0.22 to 0.54 in SCFBs. Here also porosity fraction is less in the bricks made with higher percentage of cement than low percentage. In FCBs, average porosity fraction was observed to be 0.22, which is again less than SCFBs.

3.2. Bulk density of bricks

The minimum density corresponds to the maximum volume of closed pores in the sample. Densification is a pore-filling process that occurs during the liquid-phase flow and by pore shrinkage [22]. The results of the bulk density of brick samples with different proportions of cement–fly ash and fired clay bricks are shown in Table 4. Bulk density in cement–fly ash bricks was found between 1.70 and 1.87 g/cm³. Results indicate that bulk density in cement–fly ash bricks is directly proportional to the cement percentage. In fired clay bricks, bulk density was found to be 2.11 g/cm³. Clay bricks normally have bulk density of 1.8–2.0 g/cm³ [23]. Results indicate that the bulk density of bricks meet the desired criteria.

3.3. Water absorption rate of the bricks

The water absorption rate, which refers to the weight of moisture in the pores compared to the specimen's weight, is an effective index for evaluating the brick quality. The less water penetration in the brick indicates its greater durability and resistance to the natural environment. Table 4 shows the results of the water absorption tests for various SCFBs and FCBs. The water absorption was found to be between 13.61–31.70% in SCFBs and 10.43–10.63% in FCBs. Results indicate that the bricks with higher percentage of cement have less water absorption in the case of SCFBs. FCBs show less water absorption which indicates its good durability. Only SCFBs with cement percentage of 45% and FCBs met the first-class water absorption standard (15%). No other SCFBs met even second-class water absorption standard (20%) [24,25].

3.4. Compressive strength of bricks

The compressive strength is the most important engineering quality index for building materials. The compressive strength of the SCFBs with cement percentage 30 and 45 and FCBs met the CNS 1127-R3042 standards: 150 kg/cm² for a first-class brick and SCFBs with cement percentage 15 met the CNS 1127-R3042 standards: 100 kg/cm² for a second-class brick. The results of the compressive strength of samples with different proportions of cement–fly ash and fired clay bricks are shown in Table 4. The results of compressive strength testing of the FCBs show very good results i.e. an average between 420.03 and 457.12 kg/cm² which is even higher than that of SCFBs with cement percentage of 45 (average 281.15–269.03 kg/cm²).

3.5. Acid leachability

Acid leachability tests were carried out to see the elemental migration from bricks with acids. For this purpose Al, Cu, Fe, Co, Cr, Ni, Pb, Zn and AOX analysis was done and results are presented in Table 5. The data shows that concentrations of all parameters are within acceptable limits, wherever such information is available. The metal concentrations in the leachate of different types of bricks do not follow a definite trend. But in the case of AOX, leachates of both the types of clay fired bricks have AOX concentration much lesser than that in the leachates of SCFB bricks. That may be due to the fact that during the firing significant amount of chlororganics are transformed and leave with the exhaust gases. As Demir et al. [6] concluded that pulp residue is easily burnt out from the clay body during firing.

Table 6
Characterization of leachate of water leachability test of bricks.

Sample	Al (ppm)	Cu (ppm)	Fe (ppm)	Co (ppm)	Cr (ppm)	Ni (ppm)	Pb (ppm)	Zn (ppm)	AOX (ppm)
KF15	15.2765	0.0155	0.352	ND	ND	0.0605	ND	0.12	14.57
KF30	8.306	0.023	0.2345	ND	ND	ND	ND	0.082	21.89
KF45	16.0935	0.009	0.361	ND	ND	ND	ND	0.1165	20.47
EF15	22.752	ND	0.6945	ND	ND	ND	ND	0.064	20.35
EF30	16.1865	0.0095	0.4355	ND	ND	0.052	ND	0.1755	22.66
EF45	13.352	ND	0.293	ND	ND	0.046	ND	0.088	4.66
EC	0.4135	ND	0.475	ND	ND	0.0505	ND	0.1875	1.8
KC	0.4795	ND	0.4785	ND	ND	0.0735	ND	0.1355	2.3
WF45	13.227	0.0131	0.29508	ND	ND	0.0241	ND	0.1064	ND
WC	0.4068	ND	0.4568	ND	ND	0.052	ND	0.1515	ND

Abbreviations: EF = cement fly ash bricks with membrane filtrate concentrate of extraction stage effluent; KF = cement fly ash bricks with membrane filtrate concentrate of combined bleach plant effluent; EC = fired clay bricks with membrane filtrate concentrate of extraction stage effluent; KC = fired clay bricks with membrane filtrate concentrate of combined bleach plant effluent; WF = fly ash brick with natural water; WC = clay brick with natural water; 15, 30 and 45 in sample are the percentages of cement in that particular brick.

3.6. Water leachability

Water leachability test is done to see migration of elements with water from the bricks. Heavy metals i.e. Al, Cu, Fe, Co, Cr, Ni, Pb, Zn and AOX (adsorbable organic halides) were analyzed and results are presented in Table 6. The data shows that SCFBs leachate carry much higher amount of aluminum in comparison of the leachate of FCBs. Leachate also carry much higher concentration of AOX in comparison of the leachate of FCBs, similar to trend of acid leachate. The AOX concentration was found to be more in water leachate than in acid leachate. This may be because organic halides dissociate into ions in formic and acetic acid [26], and acetic acid solution was used in acid leaching which dissociated organic halides.

Results were also compared with the brick samples made by using retentate, to the brick made by regular natural water. No significant variation was observed in the leaching of heavy metal; only variation in the AOX is said to be significant, but keeping the fact in mind that the huge amount of AOX is present in the retentate and very less proportion of that leached out experiment can be said to be successful.

Thermal treatment at 800–950 °C makes heavy metals immobilized [27]. Sorption and incorporation of heavy metals on ferrous oxide is well known and has been employed for the removal of heavy metals from the wastewater and liquid waste [28,29]. Iron oxide is principally used as adsorbent for toxic metals in the ferrox process in which ferrihydrite is formed in the slurry after precipitation of heavy metals containing ferrous hydroxide at higher pH. Ferrihydrite has a larger binding capability for heavy metals due to its larger surface area and ability to readily integrate other metals in its structure. Recently stabilization of air pollution control residue by co-precipitation with ferrous iron and subsequent thermal treatment at 600–900 °C has been reported [28]. On the basis of observation of a reduced leaching of metals, it has been described that an increase of metal binding due to thermal treatment decreases their release considerably. According to Sorensen et al. [28], ferrihydrite formed initially, is transferred in more stable and crystalline iron oxides after thermal heating, and has the potential to integrate metals in to it.

In addition, the presence of incomplete inner d-orbit of transition metals can also play a major role in their immobilization during thermal treatment. Transition metal like Fe, Ni, Mn, Zn, Cr which have an inner vacant d-orbit (d1–9) can accommodate electrons either from themselves or from other elements present in the system for the formation of complex compound. There is a high probability for them to make a strong interaction by making inter co-ordinate bonding while heating. During heating these metals are likely to be separated from their original oxide forms of the waste and apparently make inter co-ordinate bonding with each other. Once this is complete, a complex compound may form in the crys-

talline lattice of solidified product. Under this situation, separation of either metal is quite difficult [30].

4. Conclusions

After the reuse of permeate of membrane filtration in the process, retentate (concentrate) is the main liquid residue obtained from the bleaching section of the paper mill. This pollutants rich residue is usually sent to biological treatment/land filling, creating environmental concerns. Proper use of retentate obtained from paper mill effluent after membrane treatment, is achieved by using this liquid residue for brick manufacturing. Physical and chemical properties of bricks especially fired clay bricks are not adversely affected by the use of retentate. Therefore, the organic matters of the wastewater create no problems for such an application. The main advantage for the industry applying membrane filtration technology for the system closure is to save treatment cost of the concentrate, while building material industry shall be benefited by reducing fresh water consumption, while producing equivalent or better products.

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