ABSTRACT

The textile industries, after agriculture, continue to be the second largest employment generating sector in India. These industries are major consumers of fresh water, dyes, and other chemical additives such as electrolyte salt for their processing. The effluents generated from each processing step comprise substantial quantities of unutilized resources. The effluents if discharged without prior treatment become potential sources of contamination due to their several deleterious effects on the surroundings. The treatment of heterogeneous textile effluents therefore demands the application of environmentally benign technology with the water reclamation potential of appreciable quality. These features can be observed in various innovative membranes based techniques. Moreover, in the textile industry, high salinity waste streams are a challenge, urging for the resource recovery (e.g., NaCl electrolyte salt), requiring a treatment going beyond the classical filtration by e.g., reverse osmosis (RO) to produce pure water. The major problems associated with membrane process are fouling of membranes and concentrate management.

The present research is aimed at zero liquid discharge (ZLD) of textile wastewater for the maximum recovery of water at industrial scale and the brine of reusable quality on pilot scale. In the first phase of this study, a six stage pretreatment process was employed for nearly zero biological oxygen demand (BOD) reduction to eliminate the biofouling of the membrane. The sequential operation of the pretreatment process was double stage biological treatment, chemical oxidation by gaseous chlorine, coagulation and settling, activated carbon & resin filtration and ultrafiltration. The double stage ASP was optimized to achieve the maximum reduction in
BOD$_3$ by suitable hydraulic retention time (HRT), mixed liquor volatile suspended solids (MLVSS) and sludge volume index (SVI). The reduction in BOD$_3$ was achieved with 3600 mg/L MLVSS and 70 h HRT while the SVI was maintained less than 100. The chemical oxidation by gaseous chlorine was optimized to achieve the maximum reduction in chemical oxygen demand (COD) and colour removal by suitable pH, chlorine dosage and contact time. The study achieved 47% COD reduction and colour reduction of 59% at an optimum pH, contact time and chlorine dosage of 8.0, 45 min and 200 mg/L respectively. The coagulation and settling process were optimized to maximize the reduction of COD, colour and turbidity by varying the alum dosage. The study achieved a COD reduction of 17%, while colour and turbidity achieved the reduction of 83%, 90% respectively at an optimum alum dosage of 200 to 350 mg/L. The activated carbon and resin filtration process was optimized to maximize the reduction of BOD, COD and colour by varying the dosage of activated carbon and resin. The study achieved the BOD reduction of 62%, while COD and colour achieved the reduction of 32%, 83% respectively at an optimum dosage of 900 mg/L and 150 mg/L for activated carbon and resin. The UF process was studied for achieving turbidity and SDI$_{15}$ reduction by varying the turbidity. The study achieved the average turbidity reduction of 95% and silt density index (SDI$_{15}$) of less than 3 at an optimum feed operating pressure of 0.2 MPa. The wastewater characteristics such as turbidity, colour, iron, COD and BOD are primarily reduced prior to the chemical oxidation process. Silica is removed by biological treatment, coagulation and settling. Copper is removed by coagulation and settling, activated carbon filter (ACF) and resin filtration. The integrated pretreatment processes achieved the following overall reductions in characteristics such as colour 99.8%; turbidity 99.8%; copper 90%; iron 90%; silica 68.7%; COD 94.3% and BOD 99.7%. The treated wastewater was used as an RO feed in the water recovery process.
In the second phase, an integrated membrane processes (IMP) involving RO and ultrafiltration (UF) were investigated for the high permeate recovery. In the primary RO process, pretreated wastewater was used as a feed source. In the subsequent RO stages, the reject water from previous steps was used as a feed water. In the primary RO process, a brackish water membrane was used, but in subsequent RO stages, seawater membranes were used based on the total dissolved solids (TDS) in the RO feed. The feed-side pressure was increased from the primary RO stage through the tertiary RO stage, because of the increase in foulants and turbidity at these stages; however, the permeate water flux was maintained at optimum levels. All the three stages were optimized by varying the flux and operating pressure to get an optimum permeate characteristics. The results revealed that the operating pressure for primary, secondary and tertiary stages of RO were 2.3-2.5, 4.2-4.5 and 5.5-5.7 MPa, respectively, and the optimum flux obtained were 16.0, 6.0 and 8.5 L/m²h respectively to produce the average permeate TDS of 124, 230 and 550 mg/L respectively. The three stages of RO membrane were also evaluated for the concentration polarization (CP), represented as scaling potential, showed a negative LSI and S&DSI values indicating the absence of fouling supported by the SEM-EDX analysis of membrane surface of tertiary RO stage. In addition, varied Polymer-based antiscalants were employed in the secondary and tertiary RO processes for the fouling reduction and extending the membrane life. A UF treatment was employed before the tertiary RO stage, which achieved a SDI<sub>15</sub> of less than 3. The overall permeate water yield of the IMP was 92.2%, which was useful for water reuse in textile dyeing industries. Additionally, it minimizes the use of costly and environmentally stressed thermal based techniques for the conversion of tertiary RO reject into disposable solid wastes. Hence, it reduces the economic impact of waste disposal in the textile wastewater treatment.
In the third phase, concentrate management of RO process was performed for the recovery of reusable quality brine using the dual stage nanofiltration (NF) process. The bench scale study demonstrated that the primary NF membrane of molecular weight cut-off (MWCO) 1.0 kilo Daltons (kDa) resulted in the organic fouling reduction. The secondary NF membrane of MWCO 0.2 kDa was used based on the inorganic contaminants present in the feed water. The feed-side pressure applied was different from the primary and secondary NF, because the primary NF process was designed to handle organic foulants and turbidity. The secondary NF process was used for the removal of inorganic contaminants; however, the permeate flux was maintained at optimum levels. The anti-fouling and membrane life time was enhanced by using the polymer based antiscalants. ACF/Microfiltration (MF) pretreatment was carried out prior to primary NF process to achieve an SDI less than 5. The dual stage membrane process achieved brine having a pH of 5.8 and a TDS of 41,600 mg/L, with 2 mg/L of silica and 15 mg/L of total hardness, which is of sufficient quality to be reused for cotton hosiery dye bath. The SEM – EDX and X-ray diffraction (XRD) analyses were conducted to study the permeate characteristics of each process and the purity of brine recovered. The permeate and brine recovered was used for the cotton hosiery dyeing at a laboratory scale. Three dyeing tests were carried out for preparing light, medium and dark shades for cotton hosiery with increasing concentrations of brine respectively. It was observed that all the shades produced 100% colour shade and wash fastness except for a brilliant red CA light shade with 0.01 variation. The dyeing yield obtained was 92%, 94% and 98% for light, medium and dark shades respectively. The quality of brine obtained by this novel approach is on par with the characteristics of fresh NaCl.

The economical performance was studied by considering the capital and operating cost of the membrane process, concentrate disposal and the
revenues generated from the recovery. The total capital cost (TCC) for the conventional water recovery process (CWRP) and IMP are USD658400 and USD733700 respectively. The combined TCC for the IMP/ multiple effect evaporator (MEE) approach requires reduced TCC of USD899700, while the CWRP/MEE approach requires USD1075400, due to the high cost incurred for MEE. The annual TOC of the IMP/MEE approach is USD228430, while the CWRP/MEE requires USD426050 due to the higher operating cost spent towards MEE operation. The annual total revenue generated from the IMP/MEE is USD198500, while the CWRP/MEE approach generates lesser revenue, USD166000. The operational difficulties and poor quality of the condensate generated for reuse in MEE of CWRP makes unviable to consider the recovered water for economic analysis. The total payback period for the IMP/MEE is 9 years 9 months, while for the CWRP/MEE is 31 years. The CWRP approach is in viable since payback period is higher than the life of the plant. The overall economic analysis by adopting the above described method of calculation proves that increasing the water recovery in membrane process enhances the technical feasibility and economic viability of the ZLD approach. The water recovered in the IMP have been used for all the manufacturing process such as sizing, desizing, bleaching, neutralizing, dyeing, washing, soaping, fixing and softening operations and produced similar quality of the dyeing properties.

The research revealed that the textile wastewater treated using three phase system result in high permeate and resource recovery of reusable standards, which aids the textile sector aiming at ZLD.