CHAPTER 4
SEPARATION STUDIES ON MAGNETIC IRON OXIDE LOADED ACTIVATED CARBON

4.1 Introduction

One of the major unit operations in any industry is the separation process. A separation operation separates a multi component input stream into two or more output streams whose compositions differ from that of the input stream or separates a multiphase stream into its constituent sub streams. To separate a multi component stream different techniques are employed such as distillation, gas absorption and stripping, liquid – liquid extraction, fractional crystallization, dialysis and pressure – swing adsorption (Robert. P & Don, G., 1997). The criteria for separation performance are based on the product purity and the fractional recovery.

The magnetic separation comes under the type where magnetic field or gradient is used. Magnetic separation is a process in which two or more solutes are separated from each other. The primary driving force of the separation is magnetization; however there are also other forces that act upon the particles as well. These forces are centripetal force and the gravitational force (Resnick, H. & Walker, P., 1997). The controlling factors include: feed rate, velocity of the particles and magnetic field strength. Conventional magnetic separation methods have been used for a long time as a standard technique in a variety of laboratory and industrial applications, including the enrichment of low grade iron ore, the removal of weakly magnetic coloured impurities from kaolinite clay, the removal of magnetic pollutants from the stack gases from several industrial processes, the desulphurization of coal and the removal of ferromagnetic impurities from large volumes of boiler water in both conventional and nuclear power plants. The magnetic separation has now been tried in wastewater treatment as well. Many effluents contain particles, ions,
molecules, etc. possessing magnetic properties (Núñez, L. & Kaminski, M. D., 1998). The applications of magnetic separation for the efficient and economic removal of petroleum products from aqueous solutions and mixtures have been recently reported (Apblett, A. W., et al., 2001). This innovative technology of using magnetic materials to solve environmental problems, such as accelerating the coagulation of sewage (Booker, N. A., et al., 1991), removing radionuclides from milk (Sing, K. S.W., 1994), adsorption of organic dyes (Safarik, I., et al., 1995), and oil spill remediation (Orbell, J. D., et al., 1997) has gained considerable attention. A simple demonstration of the application of magnetic composites for the adsorption of water contaminants and their separation is given by Oliveira and co-workers (2004).

The non-invasive technique of separating magnetic iron oxide loaded activated carbon composites after adsorption of contaminants in solution is presented in this chapter. The main objective of this study was to find out the efficiency of separation of the prepared magnetic iron oxide activated carbon composites in a flow through system. The dependence of separation efficiency on different system parameters was studied. The dependence of separation efficiency on the gravitational force acting on a particle in a settling column, particle size of the composite samples, flow velocity of the solution and the magnetic field strength were studied.

4.2 Materials and Methods

4.2.1 Materials

(i) Adsorbent samples

The control carbon and the magnetic iron oxide loaded composite samples were used in this study. The particle size of these adsorbent samples varied from 62-88, 88-125 and 125 to 177 microns. The samples were stored in plastic containers and kept in a desiccator loaded with silica gel.
(ii) Glasswares

The study was carried out in an experimental setup made of glass. A settling column with provisions for fixing the permanent magnets was fabricated. A schematic diagram of the settling column and the separation setup is given in Figure 4.1. The settling column has a length of 14 cm and a diameter of 3 cm. The middle portion of the settling column where permanent magnets are placed has a diameter of 3.5 cm. Provisions are given in the column for the inlet and outlet of the water carrying the carbon particles. The particles retained in the column can be taken out through the tap fixed at the bottom of the settling column. The mixing of carbon particles in water was done in a 1 L glass container with a valve fitted at the bottom. This container is mounted on an adjustable stand and its height can be adjusted to deliver liquid at different flow velocities.

(iii) Instruments

Stirrer: A universal motor stirrer of type RQ – 122 (Remi Motors, India) was used for mixing the carbon samples in water.

Gauss Meter: A digital Gauss meter (Control Systems and Devices, India) was used for measuring the magnetic field strength exerted by the permanent magnets.

(iv) Permanent Magnets

Three ring type permanent magnets were used for the separation studies. Each of these magnets had an outer diameter of 5.1 cm and inner diameter of 3.6 cm. Field strength of each magnet was measured using a Gauss meter. Magnets were kept labelled and protected to prevent de-activation.
Figure 4.1 Schematic figure of the magnetic separation setup
Methods

The removal of magnetic iron oxide loaded activated carbons from the treatment trail was studied in the settling column described above. 0.5 g of the adsorbent samples was accurately weighed and added to measured volume of (1 L) of water taken in a glass container. For this study only tap water was used, as large volumes are required. The carbon particles were well dispersed in water using a stirrer rotating at a constant speed. The beaker and the settling column were connected through a flexible tube. The valve at the bottom of the beaker is then opened to release water carrying carbon particles to flow into the settling column. The carbon particles along with water rise up the settling column and over flows through the outlet of the filter assembly. The carbon particles were collected on a previously wetted filter paper (Whatman No.1). Those particles retained in the settling column were retrieved by opening the tap at the bottom of the settling column. The filter paper containing carbon particles was then dried in a hot air oven at 110 °C for 1 hour. The weight of the carbon particles collected on the filter paper was determined. The percentage retention efficiency of the process was determined using the equation:

\[
\% \text{ Retention Efficiency} = \frac{W_1 - W_2}{W_1} \times 100
\]

where, \( W_1 \) = Weight of the carbon added (0.5g)
\( W_2 \) = Weight of the carbon that flowed out of the settling column

Adjusting the head varied the flow velocity of the water that passes through the settling column carrying carbon particle. Since no attempt was made to maintain a constant water level in the storage, it was necessary to determine the average flow velocity at a particular head. The average flow velocity at a particular head was determined by measuring the volume of water discharged through the outlet tube of
the settling column for a fixed time interval when the water level in the beaker was maintained at 0.5L mark. These measurements were repeated six times and the average value was used for determining the average flow velocity of the medium.

The dependence of separation efficiency on the gravitational force and thus the settling of particles by virtue of their density were determined at zero magnetic fields. The control carbon, GAC, and the composite samples, MGAC5, MGAC7 and MGAC9 were allowed to flow through the settling column at different flow velocities. The separation efficiency due to the gravitational settling of these particles was determined by measuring the amount of particles retained in the settling column under these conditions. The influence of size of the particles on the gravitational settling was also studied at three different particle sizes of the adsorbent samples.

The dependence of separation efficiency of the composite samples on the magnetic field strength was studied at three different magnetic fields using three separate magnets. These magnets were calibrated using a Gauss meter. The field strength was adjusted by varying the number of magnets used at a time. The influence of flow velocity and particle size of the carbons at different magnetic fields on the separation efficiency of the composite samples was studied. The dependence of the flow velocity on percentage removal of the composite samples was studied by changing the hydraulic head at a constant magnetic field. Similarly the influence of particle size at a constant magnetic field on the separation efficiency was also studied by varying the size of the carbon particles.

The influence of particle size on the separation of GAC and composite samples were studied at different flow velocities and magnetic field strengths. The carbon samples had an original particle size of 125-177 microns. These particles were ground using a mortar and pestle and sieved to different size groups. The other two
size groups selected for this study were 88-125 and 62-88 microns. This study is important as activated carbons of smaller particle size, (or the powdered activated carbons) possess higher surface area and greater adsorption capacity compared to that of larger particle size (or granular activated carbon) (Metcaff & Eddy, Inc., 1991).

Results and Discussion

Separation Efficiency at Zero Magnetic Fields

The separation studies on the control sample and the composite samples (MGAC5, MGAC7 and MGAC9) were done under the influence of gravitational settling alone. This was to find out the extent of removal of the carbon particles due to gravitational settling alone in the settling column. The dependence of separation efficiency due to gravitational settling was studied for different particle sizes and flow velocities. The difference in the percentage retention of particles in the settling column for different size groups and flow velocities under the sole influence of gravitational settling are given in Figures 4.2 - 4.4. The percentage retention of carbon particles in the settling column was in the order GAC < MGAC5 < MGAC7 < MGAC9. This variation is attributed to the corresponding higher value for the density for these samples. The iron oxide impregnation on the activated carbon matrix has increased the absolute density of the composite sample and the increase is in proportion to the amount of iron oxide loaded. The values of absolute density of each sample are given in Chapter 2.

The dependence of settling of particles in the column on the size of the particles is clear from these graphs. It is clear that as the particle size gets smaller, less amount of carbon particles were retained in the column. The hydraulic thrust exerted by water carries away the smaller carbon particles. When the particle size becomes smaller the upward thrust exerted by the hydraulic pressure dominates over the
Figure 4.2 Particles retained in the settling column at zero magnetic-field at flow velocity of 5.67 cm/s.

Figure 4.3 Particles retained in the settling column at zero magnetic field at flow velocity of 7.4 cm/s.
Figure 4.4 Particles retained in the settling column at zero magnetic field at flow velocity of 8.98 cm/s.
settling velocity exerted by the gravitational force. The acceleration of the particle is related to the difference between the drag and buoyancy forces. A steady velocity, terminal velocity, is reached when these forces are balanced. The terminal velocity of the particle relative to the fluid is given by the following equation

\[ V_p = \frac{(\rho_P - \rho_L) \times g \times d_p^2}{18 \mu_L} \]

where,

- \( d_p \) = diameter of the particle
- \( \rho_P \) = density of the particle
- \( \rho_L \) = density of the medium
- \( \mu_L \) = Viscosity of the liquid
- \( V_p \) = terminal velocity of the particle

The dependence of percentage retention of particles in the settling column was studied using three different particle sizes. It is evident that as the particle size decreases the influence of iron oxide loading on the retention efficiency was reduced. The percentage of particles retained in the column is drastically reduced at higher flow velocities.

**Dependence of Retention Efficiency of Composite Samples on Iron Oxide Loading**

The dependence of retention efficiency of composite samples under different flow velocities and magnetic fields on iron oxide loading was studied. The variation in the percentage retention of particles in the settling column under different flow velocities and magnetic field strengths are given in Figures 4.5 - 4.13. The study shows that the decrease in particle size reduced the percentage retention of carbon particles in the settling column. But the percentage loading of magnetic iron oxide on
Figure 4.5 Retention vs Flow velocities for different particle sizes for MGAC5 at a field strength of 49 G.

Figure 4.6 Retention vs Flow velocities for different particle sizes for MGAC5 at a field strength of 99 G.
Figure 4.7 Retention vs Flow velocities for different particle sizes for MGAC 5 at a field strength of 147 G.

Figure 4.8 Retention vs Flow velocities for different particle sizes for MGAC 7 at a field strength of 49 G.
Figure 4.9 Retention vs Flow velocities for different particle sizes for MGAC 7 at a field strength of 99 G.

Figure 4.10 Retention vs Flow velocities for different particle sizes for MGAC 7 at a field strength of 147 G.
Figure 4.11 Retention vs Flow velocities for different particle sizes for MGAC 9 at a field strength of 49 G.

Figure 4.12 Retention vs Flow velocities for different particle sizes for MGAC 9 at a field strength of 99 G.
Figure 4.13 Retention vs Flow velocities for different particle sizes for MGAC9 at a field strength of 147 G.
the composite samples exerted a significant influence on the retention efficiency under different magnetic field strengths even when the particle size was reduced. The percentage retention obtained for MGAC5, which has the least iron oxide loading, varied between 60-100 % under different flow velocities and magnetic fields. The variations are given in Figures 4.5 - 4.7. The retention for MGAC7 varied from 80-100 % at different flow velocities and magnetic field strengths. Figures 4.8 - 4.10 gives the variation in particle retention at varying flow velocities and magnetic fields. More than 99% retention of the particles was achieved for MGAC9 for all the three particle size ranges. This was evident from Figures 4.11- 4.13. The overall retention capacity increased with magnetic iron oxide loading.

**Dependence of Retention Efficiency of Composites on Magnetic Field**

The dependence of separation efficiency of composite samples MGAC5, MGAC7 and MGAC9 on the magnetic field applied is drawn from Figures 4.5 - 4.13. Magnet number one, two and three has an effective magnetic field of 49, 50 and 48 Gauss respectively. It can be seen from these plots that higher the field applied greater the percentage of particles retained in the settling column. The influence of varying magnetic strength is more evident in the case of lower iron oxide loaded samples, MGAC5 and MGAC7 where the percentage retention depends on the magnetic field applied. For higher magnetic iron oxide loading (MGAC9) the variation in the magnetic field did not influenced the retention capacity significantly.

The effect of particle size on the retention efficiency at different magnetic fields was also studied. It can be seen from these plots that the influence of particle size on the separation efficiency was more visible for composite samples, MGAC5 and MGAC7. When the size of particles was decreased, the retention efficiency was
reduced. It is observed that irrespective of particle size, the retention capacity for MGAC9 was close to 100% at all magnetic field strengths.

When the flow velocity was increased from 5.67 cm/s to 8.98 cm/s under different magnetic fields, a reduction in the retention efficiency was observed for MGAC5 and MGAC7. The reduction in the separation efficiency with increase in flow velocity is more predominant in the case of MGAC5 compared to MGAC7. And the influence of flow velocity on the composite sample, MGAC9 was only marginal under different magnetic fields. This suggests that for a higher magnetic iron oxide loading the flow velocity has little influence on the removal of particles from the stream at different magnetic fields.

**Dependence of Retention Efficiency on Flow Velocity**

The influence of flow velocity of the medium on the retention efficiency of carbon particles was studied at different particle sizes and magnetic field strengths. The dependence of retention capacity on the flow velocity in the settling column is shown in Figures 4.5 - 4.13. When the flow velocity of the medium was increased from 5.67 cm/s to 8.98 cm/s, the percentage retention decreased. The reduction was more significant for MGAC5. For higher magnetic iron oxide loaded carbons the influence of flow velocity on retention capacity was marginal and outweighed by the intensity of applied magnetic field.

The changes in retention capacity with particle size are significant at higher flow velocities. The capacity decreased with increase in flow velocity and decrease in particle size. Particles with smaller sizes are easily carried out of the settling column at higher velocities. However, under the influence of an applied magnetic field the influence of flow velocity on carbon particles of different size groups is reduced significantly. Separation efficiency of composite sample MGAC9 of different particle
sizes at varying flow velocities was not reduced significantly under the influence of varying magnetic fields.

The dependence of retention efficiency on the flow velocity and varying magnetic fields for each composite sample is given in Figures 4.5 - 4.13. The percentage reduction in the retention capacity varied as a function of applied magnetic field. An increase in the applied magnetic field reduced the dependence of separation efficiency on the flow velocity. But the gain in the separation efficiency due to higher applied magnetic fields against the flow velocity is felt more at higher iron oxide loading.