

CHAPTER 3. ACTIVATED CARBON COLUMNS PLANT DESIGN

Maybe, the first question that we have to ask ourselves is related to the decision of an adsorption process using activated carbon for the removal of micropollutants is efficient. The theory says that the adsorbability of an organic molecule increases with increasing molecular weight and decreasing solubility and polarity. This means that high molecular weight compounds with low solubility, such as most pesticides, are well adsorbed, so the first idea is plenty justified.

In this design, some aspects were taken into account.

3.1. ACTIVATED CARBON CHOICE

Firstly, it's important to remember that activated carbon can be used in different forms, basically, as powdered or granular. GAC (granular activated carbon) is utilised in drinking water treatment by installing it in a fixed bed adsorber. Water is passed through the adsorber containing the activated carbon which adsorbs organic compounds, purifying the water, while PAC (powder activated carbon) is used in a totally different way. It's added to water, mixed and then removed at the decantation or filtration stage. The aim of this experiment is to determine breakthrough behavior of micropollutants in activated carbon columns so the decision of using GAC instead of PAC is obvious.

The next step is the selection of a granular activated carbon.

Chemviron carbon was chosen due to it is usually used in Belgium water companies.

There are a number of criteria that should be considered in this selection.

GAC for this purpose needs a pore structure to allow the adsorption of a wide range of organic micropollutants, being not so important the adsorption of natural organic matter (tap water was used in the experiments). The GAC must also possess a suitable amount of transport pores which allow the molecules to be transported to the adsorption site. The adsorption capacity for drinking water applications is very difficult to quantify by laboratory evaluation. Parameters such as the iodine number indicate the overall porosity of the carbon, but cannot be used to estimate the performance in drinking water applications. The design should be based on experience and references, so this was the idea for the selection.

FILTRASORB 400 is the most widely used and effective GAC for pesticide removal with excellent performance for a wide range of pesticides.

3.1.1. Filtrasorb 400.

3.1.1.1. Description.

FILTRASORB carbons are produced by steam activation of selected grades of bituminous coal that have first been pulverised then agglomerated.

It has high adsorption capacity and a high number of transport pores. This gives the carbon a greater selectivity for the removal of micropollutants in the presence of natural organic matter.

3.1.1.2. Features.

Agglomerated coal based granular activated carbons have several properties, which explain their superior performance in a wide range of applications:

- Produced from pulverised blend, results in a consistent high quality product.
- The activated carbon granules are uniformly activated throughout the whole granule, not just the outside. This results in excellent adsorption properties and constant adsorption kinetics.
- High mechanical strength of the coal based carbon gives excellent reactivation performances.
- Agglomerated coal based carbon are suitable for multiple reactivations compared to the other base materials such as peat and wood.
- The agglomerated structure ensures rapid wetting, so there is no remaining floating material.
- Carbon bed segregation is retained after repeated backwashing, ensuring the adsorption profile remains unchanged with time and therefore maximising the bed life before breakthrough.

3.1.1.3. Selection

Two different activated carbons were taken into account, FILTRASORB 300 and 400. They have a typical effective size range of 0.9mm and 0.7 respectively. In general, the smaller the granule size, the better the adsorption performance, thus FILTRASORB 400 should be more interesting for all

applications. The principle limitation is related with the pressure drop. It was checked that it was not too high, so the final choice was FILTRASORB 400 . If it hadn't been the case, FILTRASORB 300 would had been selected.

3.1.1.4. Properties

The most important specifications and properties of the selected activated carbon are resumed in the next tables. Some of them are extremely important for the columns design.

TABLE 3.1. FILTRASORB 400 SPECIFICATIONS

SPECIFICATIONS	F400 12x40
Iodine Number, min., mg/g	1050
Methylene Blue Number, min.	260
Abrasion Number, min	75
Moisture Content, as packed, max., %w/w	2
Effective Size, mm	0.6-0.7
Mesh Size, US Sieve Series	12x40
> 8 mesh (2.36mm), max. %	-
> 12 mesh (1.70mm) max. %	5
< 30 mesh (0.60mm), max. %	-
< 40 mesh(0.425mm), max. %	4

TABLE 3.2. FILTRASORB 400 TYPICAL PROPERTIES

TYPICAL PROPERTIES	F400 12x40
Backwashed and drained Bed Density., kg/m ³	425
Floating Content, max., %w/w	0.1
Surface Area, (N ₂ BET method) m ²	1050
Mean Particle Diameter, mm	1.0
Uniform Coefficient	1.7
Phenol loading at 1 mg/l, DIN 19603, %	5.2
Detergent (TPBS) loading at 1 mg/l, mg/g	200
Atrazine loading at 1 µg/l, mg/g	40
Toluene loading at 1mg/l, mg/g	100
Trchloroethylene loading at 50 µg/l, mg/g	20

3.2. ACTIVATED CARBON COLUMNS DESIGN

Once the activated carbon that is going to be used is known, it can be thought about the columns design. In this situation, the design was developed following several recommendations found in the bibliography and typical design parametres for FILTRASORB 400 installed for the treatment of surface water. Furthermore, once the definitive design was obtained, dimensions could be compared with other columns that work in similar situation so the rule that says “alternatively, the design should be based on experience and references” was followed.

3.2.1. Some important ideas and typical design parameters

- Usually, full scaled granular activated carbon beds are from 1.0 to 10 meters in depth and from 0.3 to 4.0 meters in diameter. In a laboratory the diameter of the columns can be scaled down to 50 mm; a smaller diameter should not be selected in order to avoid excessive wall effects.
- To avoid channeling the minimum column diameter should be at least 50 times the particle effective size.
- Ratio activated carbon bed/column diameter ~ 10 .
- The backwashed and drained density is the parameter used for sizing of adsorption equipment and determining the weight of carbon required for drinking water applications (thus, in case of drinking water, the general term bed density -referring of the density of the bed when the carbon is installed in an adsorber- is the backwashed and drained density as the bed will be segregated).
- The main design parameters of a system are the contact time and linear velocity.

$$\diamond \text{ Contact Time (minute)} = \frac{\text{Carbon volume (m}^3\text{)} * 60(\text{min/h})}{\text{Flowrate (m}^3\text{/h)}}$$

$$\diamond \text{ Linear Velocity (m/h)} = \frac{\text{Flowrate (m}^3\text{/h)}}{\text{Surface area (m}^2\text{)}}$$

- The superficial contact time is typically between 6 and 30 minutes, though lower and higher contact times are utilised in a lot of situations. For a fixed flow, the volume of GAC and therefore size of the adsorbers can be calculated. That is, if you doubled the contact time, you double the volume of GAC.
- Talking about the other important parameter, it's important to said that the typical design value of linear velocity should be between 5 and 20 m/h.

3.2.2. Calculations.

Firstly, it was necessary to estimate some parameters.

Due to the activated carbon effective size was known, the column diameter was the first parameter to be estimated. Following the rules mentioned above the value decided was 5.0 cm. A smaller diameter should not be selected in order to avoid excessive wall effects and this number is around 70 times the particle effective size so we can be sure that the channeling is avoided.

Once the diameter is determined, other column dimensions can be calculated:

- The area is calculated taking into account that the column section is circular.

$$Area = \frac{\pi * D^2}{4} = 19.6 \text{ cm}^2$$

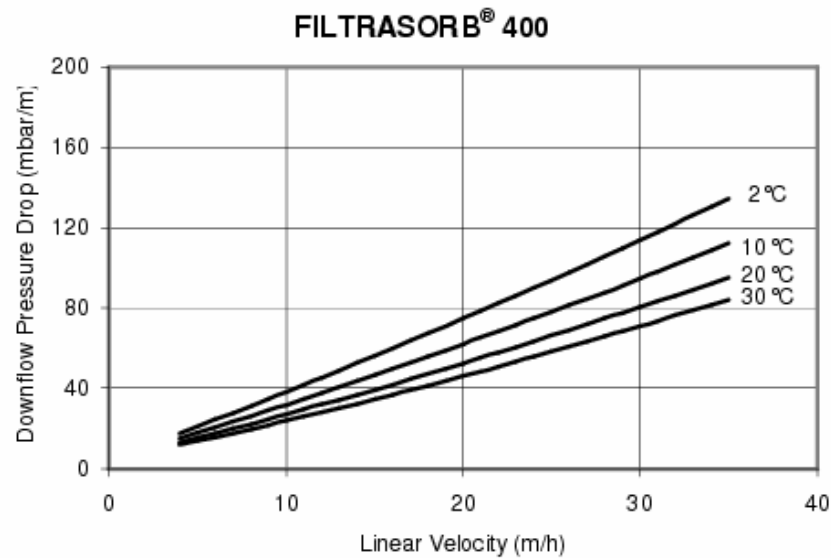
- The height of carbon bed is going to follow the relation $H/D \sim 10$, so this height was decided to be 55 cm.

The other parameter that we have to estimate is the linear velocity. In this decision, the design is based in references (obtained from Filtrasorb 400 typical values) and experience (comparing with similar situations searched in different bibliography). A linear velocity of 10 m/h can be a reasonable value that also must avoid excessive pressure drop in the bed.

The next step is to define the height of the column. Pressure drop in the column has to be known for calculating it. This calculation involves two different pressure drops:

- Pressure drop in a backwashed and segregated bed.
- Pressure drop in the discs.

For the first one, a typical pressure drop curve for backwashed and segregated bed is used. This information is provided by Chemviron Carbon. For this application we are using Filtrasorb 400, the linear velocity is 10 m/h and the operation temperature is 15°C approximately. Using these data and figure 1, the downflow pressure drop obtained is 30 mbar/m. Due to our bed is 55 cm high, the total pressure drop in the activated carbon bed is 16.5 mbar.

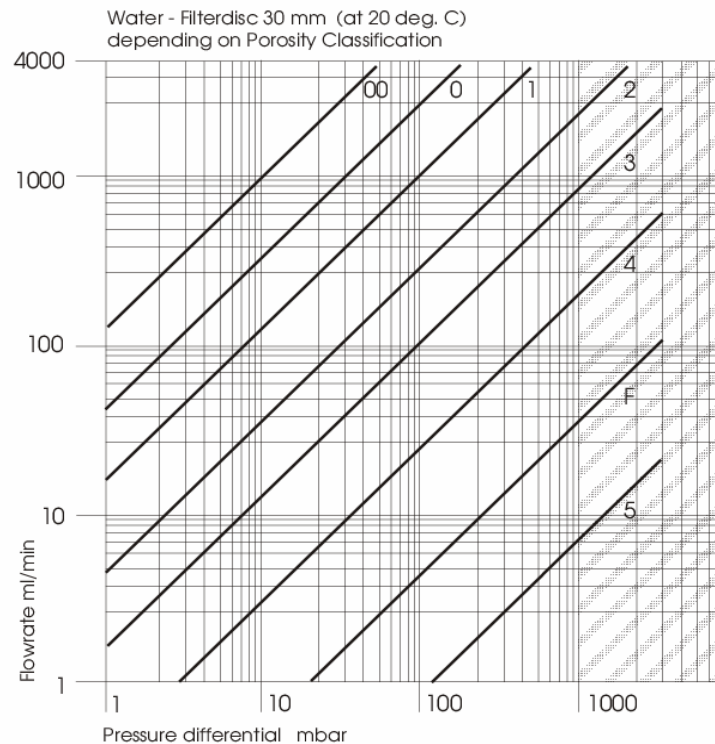
FIGURE 4.1. TYPICAL PRESSURE DROP CURVE FOR A BACKWASHED AND SEGREGATED BED

Secondly, it was necessary to calculate pressure drop in the filter disc, that is going to support activated carbon bed in the column. This is a 50 mm filterdisc.

For calculate this number, figure 2 provided by Robu has been used. The flow rate is 333.3 ml/min and curve 00 is our situation. Using this curve, pressure drop obtained is 3.5 mbar. But this number is for a 30 mm disc. In our case, we have to correct it using a 2.5 factor (table 4.3), like is explained in Robu's guide. So, the real pressure drop is 8.75 mbar.

TABLE 3.3. FACTOR ACCORDING TO FILTER DIAMETER

DIAMETER	FACTOR
10	0.13
20	0.55
30	1.00
40	1.50
50	2.50
60	4.30
90	6.80
120	9.70
175	15.00
200	16.30
250	17.40
300	25.00
350	26.70
400	30.60
450	32.50

FIGURE 3.2. PRESSURE CALCULATION VITRAPOR FILTERDISCS

To sum up, pressure drop in the column is 25.25 mbar, that can be translated into 25.25 cm.

Now, it's possible to determine the total column height. Taking in account the bed height, the pressure drop and some place for taking samples and safety reasons, each column is designed as 1 meter height.

The columns are completely dimensioned, and the next step is to calculate the operating conditions and the weight of activated carbon in each column.

Operating conditions.

For obtaining the flow rate, the expression cited before has to be used:

$$\text{Linear Velocity (m/h)} = \frac{\text{Flowrate (m}^3/\text{h)}}{\text{Surfacearea (m}^2\text{)}}$$

- *Linear Velocity = 10 m/h*
- *Surface area = 19.6 cm²*

Thus, flow rate is 19.6 l/h. In the real situation this flow will be 20 l/h, so linear velocity will change, but not too much (10.2 m/h).

The other important operating parameter is the empty bed contact time (EBCT), that can be calculated using:

$$\text{Contact Time (minute)} = \frac{\text{Carbon volume (m}^3\text{)} * 60(\text{min/h})}{\text{Flowrate (m}^3\text{/h)}}$$

where,

$$\text{Carbon volume (each column)} = 1.078 * 10^{-3} \text{ m}^3$$

$$\text{Flow rate} = 1.96 * 10^{-2} \text{ m}^3/\text{h}$$

So EBCT is 3.3 minutes in each column. Comparing to other columns with similar characteristics and according to recommendations is a reasonable number.

Finally, the activated carbon weight that is going to fill each column must be known.

$$\text{Weight} = \text{backwashed and drained density} * \text{carbon volume}.$$

where,

$$\text{backwashed and drained density} = 425 \text{ kg/m}^3 \text{ (carbon data)}$$

$$\text{carbon volume} = \text{area} * \text{bed height} = 1078 \text{ cm}^3$$

so, 0.485 kg of activated carbon must be used in each column.

In the next table, a resume of all these important parameters is shown.

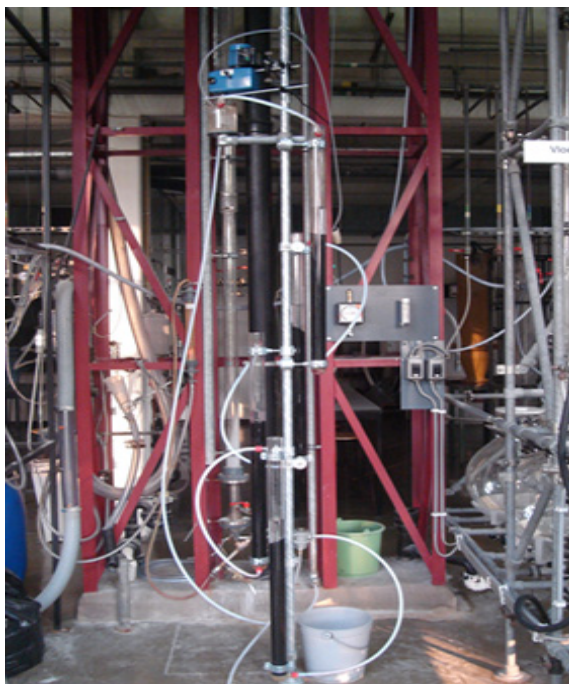
TABLE 3.4. COLUMNS DESIGN RESUME

ACTIVATED CARBON	Filtrasob 400
Effective Size	0.7 mm
Backwashed and Drained Bed Density	425 Kg/m ³
Volume	1078 cm ³ (each column)
Weight	0.46 Kg (each column)
COLUMN	
Diameter	5.0 cm
Area	19.6 cm ²
Height of carbon bed	55 cm
Height of column	1 m
OPERATING CONDITIONS	
Flow	20 l/h
Linear Velocity	10 m/h
EBCT	3.3min

3.3. DESIGN OF OTHER ELEMENTS IN THE PLANT

Once the columns were dimensionate, other elements of the plant were designed. Firstly, it is important to consider that the columns were placed in a special configuration. They were built as it is shown in the picture in a compact way and instead of needing 5 meters high to place them they were built in a 2 meters and a half place. Conections were made of plastic tubes. Figure 3.3 shows activated carbon columns.

FIGURE 3.3. ACTIVATED CARBON COLUMNS.



Above the top of the first column there is a mixing system that is used to mix properly two flows. Its volume is 800 ml and the flow that receive is about 20 l/h so its residence time is 2.4 minutes, time enough to mix the two flows of 19.5 and 0.5 l/h. Its regulation is explained below

FIGURE 3.4. MIXING SYSTEM



3.3.1. The flow regulation problematic.

It was extremely important to be sure that the pesticide concentration that was going to be fed to the granular activated carbon column was constant and the one that was previously stipulated (0.1 ppm). For this reason a special attention was paid to the flow regulation systems and a extensive discussion is developed in this section.

Two different currents were mixed to obtain the final flow of 20 l/h with a 0.1 ppm concentration. One of them was a current of 19.5 l/h of tap water and the other corresponded to the concentrated solution (4 ppm) of atrazine in distilled water and its flow was of 0.5 l/h. So it was necessary to control two different flows. Two rotameters were used to control both flows but each time that a flow needs to be controlled the pressure of the current must be controlled too. If the pressure suffers some variations, the flow is going to be altered as well. So the importance of the constant pressure is justified. If this constant pressure cannot be obtained, someone has to be permanently taking care that the flow is constant opening or closing the rotameters valve.

To control the pressure of the tap water flow a manometer was installed before the rotameter so the pressure of the current that pass through the rotameter is always constant although variations in the supply system could appear. This is necessary due to these variations that have been mentioned and usually occur. The manometer provided a constant pressure of 2.5 bar. This pressure cannot be too similar to the average pressure (4-5 bar) because it is not possible to control it correctly in this situation and it cannot be too low because it is not possible to reach the required flow in the rotameter with such a low pressure.

FIGURE 3.5. DETAIL OF MANOMETER TO CONTROL PRESSURE IN TAP WATER FLOW



Pressure control of the concentrated solution and its reservoir design. Principle of Mariotte's bottle.

The concentrated solution flow was quite small and variations in its delivered pressure could be translated into big flow differences. So it was really important to design a system that could offer a constant pressure. Pressure control and the reservoir design had to be treated together. It was necessary to store the concentrated solution in big reservoirs with enough capacity to let installation work for several days without any personal requirement. Two reservoirs of 45 litres (capacity of both) approximately were considered as a good option. The required supply of this solution was 0.5 l/h so 12 litres were necessary each day. The installation could operate for almost 4 days with these reservoirs. Their construction was based on Mariotte's bottle principle that will be explained later. Use of pumps for feeding the pesticide solution was tried to be avoided due to the small flow that was required. Valves were also avoided. The best option was to place the reservoirs in the second floor of the basement and make use of the height difference between the reservoirs and the mixing point of the two currents, so no pumps were necessary. The constant pressure was provided using Mariotte's bottle principle and valves were avoided with these construction. Some theoretical ideas are explained below.

It is well known that if we have an open air reservoir that is discharging a liquid, the discharged pressure decreases at the same time that the liquid level is diminishing. The reason is that the surface of the liquid is at atmospheric pressure and the bottom of the reservoir (discharged point) is at atmospheric pressure plus ρgh . The height (h) decreases during the liquid supply so the

pressure does it as well. Thus, the solution pressure across the rotameter gets lower and the flow lessens as well. Someone has to stay permanently in the installation to control and regulate the rotameter valve. This option is not practical. To avoid this problem Mariotte's bottle is a good alternative.

Mariotte's bottle is a device that provides a constant pressure that will deliver a constant rate of flow from closed bottles or tanks. Its name is due to Edme Mariotte (1620-1684) a french physicist that studied it. The design was first reported by McCarthy (1934). A stoppered reservoir is supplied with an air inlet and a siphon. The pressure at the bottom of the air inlet is always the same as atmospheric pressure; for if it was greater, air would not enter; and cannot be less as it is in contact with the atmosphere. If the entrance to the siphon is at the same depth, then it will always supply the water at atmospheric pressure and will deliver a flow under constant head h , regardless of the changing height of water within the reservoir. This apparatus has many variations in design and has been used extensively whe a constant water pressure is needed.

One of the mentioned variations is the bottle that was used in the instalation but the main idea is the same. Pressure at point A is always atmospheric pressure and it is extremely important to "prepare" the bottles to be sure that this is the pressure (It will be explained later). It doesn't matter the liquid level into the tank because variations in this level just influence in point B pressure ($P_B = P_A - \rho gh$). If liquid level decreases P_B would do it as well. The discharged pressure stays constant (atmospheric pressure) and liquid level pressure (point B) diminishes during operation. When we have an open air reservoir the situation is the opposite: Liquid level pressure is constant (atmospheric pressure) and the discharged pressure decreases during the discharge. Some ideas to better understand these bottles are explained below:

There is a tube that conects the bottom and the top of one of the bottles. The aim of this tube is to control the liquid level into the bottles. Due to this principle the liquid level in the small tube is the same as the liquid level into the tank.

Both bottles can be considered as one due to they are connected following the connected vessels principle. They are connected with small white plastic tubes in the bottom and in the top of the reservoirs (the liquid and the gas phase). So they work as one tank, the liquid level is the same in both and pressure distribution is identical.

It is extremelly important to explain and understand the importance of a correct filling of the reservoirs. For filling them it is necessary to open the valves that are in the top of them. When these valves are opened the Mariotte's bottle principle is not governing the situation. The bottles start to operate as an open air reservoir. The small tubes that connect the valves with the bottom of the tanks are filled with the solution and the liquid level in them is the same as it is into the bottles. So, when the tanks are filled and the valves closed, the small

tubes have some solution into them and the pressure at the end of these tubes is not the atmospheric pressure, it is the atmospheric pressure plus ρgh , where h is the liquid level height in the tubes (the same than the liquid level in the tanks). Consequently, the pressure at the bottom of the tanks is not the atmospheric and obviously the discharged pressure is not atmospheric neither. The pressure is going to decrease until some liquid has been evacuated. The atmospheric pressure will be reached and since that moment it is going to be constant. To avoid this temporary regime an important connection was constructed. It is the plastic white tube that connects the bottom of the tanks with the atmosphere. Due connected vessels principle, the liquid level in it at the beginning (when reservoirs are just filled) is a little bit less than it is in the bottles and the same than it is in the small tube inside the reservoirs (remember that both are at atmospheric pressure and the pressure in the tanks is lower than the atmospheric). But it is necessary that liquid level in this plastic tube is at the same level than the end of the inside tube to be sure that point A is at atmospheric pressure. Using different words, I want that there is no solution into the inside tube because it means that the pressure at the end of it is the atmospheric (and the most important thing, it is going to be constant). Due to there is a connection in a point a little bit lower than the end of the tube that connects it with the atmosphere, I can realise that the tube is not containing any solution when the liquid level in the outside tube (white plastic tube) is at the bottom of the controlling level tube (the same than the end of the inside tube). There is another simple way to be sure that the situation is the one that I want (constant atmospheric pressure at the discharge point). Once that the reservoirs are filled and valves are closed, I used the outside tube to evacuate the necessary solution to reach the initial situation. While the liquid is evacuated, the level decreases in the outside and inside tube at the same time. It can be understood that bubbles start to form when the inside tube is totally empty, and it can be realised due to sound of the bubbles into the tank. At this moment the Mariotte's bottle principle starts to govern the operation, the level stays constant in both tubes, the pressure is the atmospheric and bubbles are continuously forming at the end of the inside tube. The amount of liquid that is evacuated is the same amount that I pointed out when I said that some liquid was necessary to be evacuated to reach the permanent regime.

FIGURE 3.5. MARIOTTE'S BOTTLE.

3.4. ACTIVATED CARBON PRETREATMENT.

It could be realised the importance of a good preparation of the column before starting the experiments.

One of the most common errors in adsorption column test work which lead to poor results is that the carbon is not degassed prior to the adsorption test. When this is not done, air pockets form in the column and result in channelling, high pressure drop and premature breakthrough of adsorbate.

The carbon should be wetted prior to being placed in the test columns. Indeed it is extremely difficult to remove air pockets from the packed carbon bed and contrary to popular belief, backwashing the carbon at low rates does not remove air pockets.

The first time It was thought that the carbon was already pretreated so the columns were just filled according to the 55 cm height that It had been designed. When the column started running, the pressure drop was too high and the liquid level rapidly increased so the experiment had to be stopped.

Then It was thought about the necessity to backwash the granular activated carbon to segregate the bed. When the granular activated carbon is backwashed (that is an upward flow of water) the fine carbon particles and air are removed and the bed is segregated. Segregation is where smaller granules

are pushed to the top of the bed while larger granules migrate to the bottom. This results in a lower density, typically 85 % of the apparent density. This means the height of carbon in an adsorber after filling will increase after the initial backwash. So the column was filled taken into account the 15 % expansion that the bed was going to experiment after the backwash. The final height was the 55 cm that was designed. When the column was restarted with this new pre-treatment, it seemed to work properly and the pressure drop coincided approximately with the 25.25 cm that were calculated in the column design. Height was measured with a rule and it was quite exact. But after some hours running the column with a 20 l/h flow of tap water (it was just to test the hydrodynamic behaviour of the column) the level started to rise continuously.

After these two attempts It was thought about degassing the carbon. The amount of granular activated carbon required to fill each column was determined as follows:

$$\text{Weightcarbon / column} = V \times AD$$

where:

V = volume of carbon in column.

AD = apparent density of carbon.

So the weight of carbon is 0.46 kg in each column.

The required amount of granular activated for each column was weighted into a suitable container and wetted by soaking at ambient temperature for 24 hours. The degassed carbon is charged to the columns as a slurry in small increments, keeping a thin layer of supernatant liquid above the carbon during charging. This is best accomplished by filling the column one third full with the wetting liquid before starting to charge the carbon slurry.

After this pre-treatment the column responded in a similar way that it did the second time. There was not a big difference between last two options. So it was concluded that the problem was not related to the preparation of the column. It could be checked again that the pressure drop design was well calculated due to the liquid level above the carbon bed was the 25 cm that it had been predicted in calculations and it stayed like this for 6 or 7 hours. But after this time, the level started to rise again and it didn't stop until the flow was reduced.

3.5 OTHER DECISIONS

The problem mentioned before was very important due to experiments could not be developed properly. After thinking about different options, it was thought that tap water could contain air bubbles that provoke this anomalous behaviour. Water in supply system is supersaturated and then is decompressed forming air bubbles in it. Hence anomalies observed into the column could be due to these air bubbles adsorption on the activated carbon bed. The effect is not realizable at the beginning of hydrodynamic tests due to just a small amount of solution has passed through the column so a little amount of bubbles had been adsorbed. It is because of it that pressure drop is the same that was considered in calculations. When the column has been running for a long time a greater number of air bubbles has been adsorbed so their effect on pressure drop is considerable and it is continuously growing. It was a dramatic problem due to experiments were long time consuming.

To solve the problem the option was to build a degaser before the current was fed into the column as it was explained in plan design chapter. By this element air bubbles from the tap water would be removed so their presence in the column would be avoided.

The deagaser was built using a 30 cm diameter and 1 meter high plastic reservoir in which ceramic rings were introduced acting as filling material. Tap water is introduced from the top of the reservoir and leaves it from the bottom, passing through the whole bed.

It was incorporated into the plant and the operation seemed to be stable without pressure drop problems for some days. But after several days of operation, the problem started again. It plenty justified that the problem was caused by air bubbles in tap water because the degaser was saturated and air bubbles were eliminated from degaser if it was beaten. If top of the carbon bed was touched with a tube, air bubbles were also removed.

The new problem was to consider whether the degaser was saturated or air bubbles needed more time to grow up and be removed from the degaser. A new reservoir was built before the other and experiments were re-started. There was no problem using this new design.

There are two degaser pictures below.

FIGURE 3.6. DEGASER. FRONT VIEW.



FIGURE 3.7. DEGASER. VIEW



3.6. PROBLEMS WITH ROTAMETERS

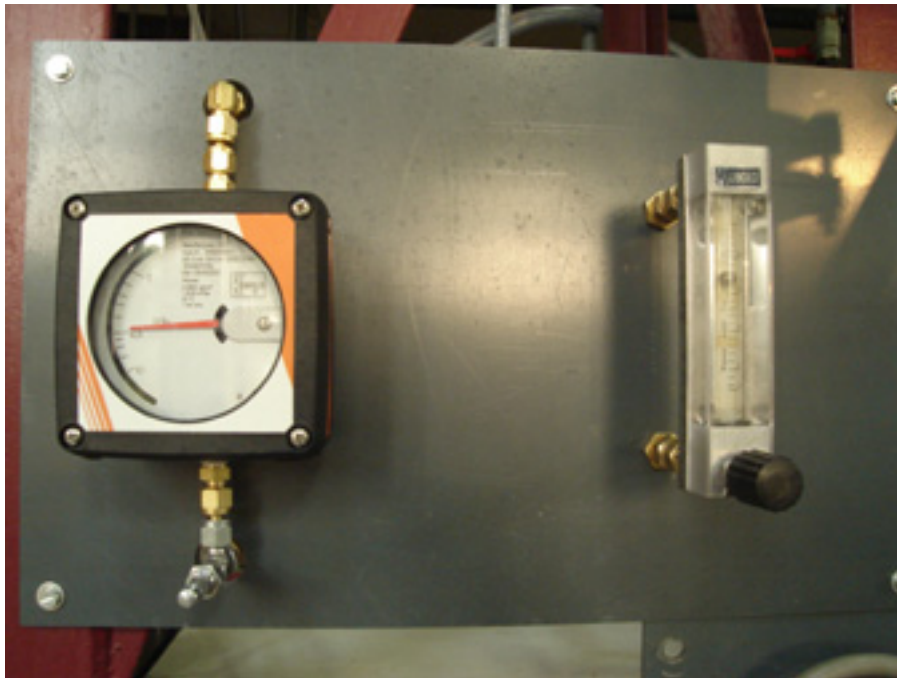
Due to air bubbles in water mentioned before, rotameters showed different values than those ones that were fixed as set points after some hours running. This is a new evidence that air bubbles were formed in tap water when

supersaturated water was decompressed. When needle pointed a higher value, rotameter was softly beaten and it went back to the set point.

Concentrated solution was prepared with distilled water instead of tap water and rotameter problem in this current disappear, fact that is a evidence of what was mentioned before. Tap water rotameter continued pointed a wrong flow but it could be tested that the real flow was the proper one. Analyzing atrazin influent concentration was used to be sure of it.

Rotameters are shown in figure 3.8

FIGURE 3.8. ROTAMETERS.



As can be realized in this chapter, the design of the plant was a complex work, that involved a lot of problems and was a long time consuming activity. Finally, plant designed was finished and it can work without problems for a big number of experiments.