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DISINQUINAMENTO DELL'ARIA INDOOR: PROVE DI EFFICIENZA DI FILTRI ELETTROFILATI

INDOOR AIR QUALITY CONTROL: TESTING OF ELECTROSPUN FILTERS

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Abstract:

Il lavoro di tesi ha riguardato la definizione di un metodo per testare l'efficienza disinquinante di filtri elettrofilati composti da Acido Polilattico (PLA- Polylactic acid or polylactide) e ciclodestrine nei confronti di particolato e Composti Organici Volatili (VOCs).

L'intento dell'intero operato è stato quello di porre una base progettuale su cui andare poi a lavorare variando alcuni parametri fondamentali quali tempo di esposizione, concentrazione degli elementi inseriti,….
La filiera di processo si compone di due modelli a box non collegati tra loro utilizzati rispettivamente per la generazione e il ricircolo, tramite una ventola interna, dell'inquinante modello utilizzato per la valutazione dell'efficienza di rimozione del filtro in esame.

Le tappe fondamentali sono state:
- assembramento dell'intera filiera di prova con organizzazione del laboratorio per ogni test da eseguire
- valutazione dei filtri elettrofilati messi a disposizione (ognuno con una composizione specifica)
- esecuzione del test vero e proprio con la conseguente osservazione dei risultati ottenuti dopo aver impostato i vari parametri di processo
- elaborazione grafica dei risultati

Il metodo di prova, esposto con dovizia di particolari nell'elaborato di tesi, ha consentito l'individuazione delle migliori condizioni per una valutazione accurata dell'efficienza di rimozione dei filtri con la possibilità di osservarne il comportamento durante tutto il processo, tenuto costantemente in condizioni "controllate".

INDOOR AIR QUALITY CONTROL: TESTING OF ELECTROSPUN FILTERS

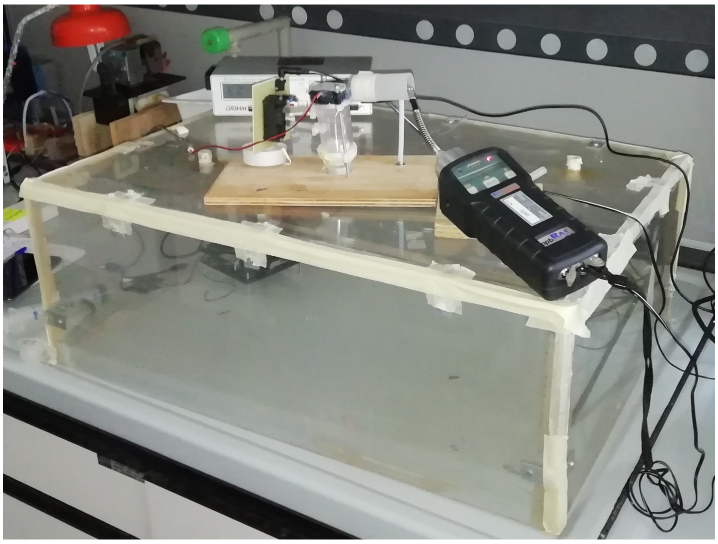
1. INTRODUCTION

1.1 INDOOR QUALITY ISSUE

Air pollution is one of the biggest and more discussed problem nowadays that involves all the entire world. The surrounding environment is composed by some compounds: especially in indoor spaces, it is possible to find a variety of them quite different from what we can find outdoor because they come from smoking, products to clean the house, room perfumers and domestic appliances as examples. In addition to that ones already described, the air exchange between indoor and outdoor spaces increases pollution inside.
Risks connected to air pollution are serious for people because of exposition in time and this can have bad effects for human health. In addition to this aspect, it is important to notice that people all over the world spend their time especially inside (work, house, gym,…) and not outside (parks,…) so problems connected to indoor air pollution are becoming worst and worst. Some researches show that pathologies like asthma and allergies (in children as an example) are strictly linked to a prolonged exposition to confined spaces air and also performance (speaking about work or study) aren't optimal.
The OMS classifies VOCs (Volatile Organic Compounds, the root cause for bad indoor air) in 4 groups based on boiling point and on how they are volatile (a little or too much) while the ECA (European Concerted Action) bases the classification on the chromatographic retention (which involves dimethylpolisilossano).

To give an idea about how VOCs could be dangerous for human health, it is necessary to study the indoor space (in which the research has to take place) to know specificities about it, such as activities, how long people stay in this confined space and also about dimensions.
AIR ANALYZER

Our research and project was all about testing new technologies to remove air pollution from indoor spaces, using some filters made up with PLA and Cyclodextrins which help purifying air in closed spaces and prevent human health from becoming worst. Our goal was to measure the efficiency of each filter in front of small particles (PM) and volatile compounds (VOCs), which came from incense burning and toluene inside the model box that represent the indoor space, using instruments like the "Grimm" (for PM) and the "ppb RAE" (for VOCs).



The "model box" used in our tests

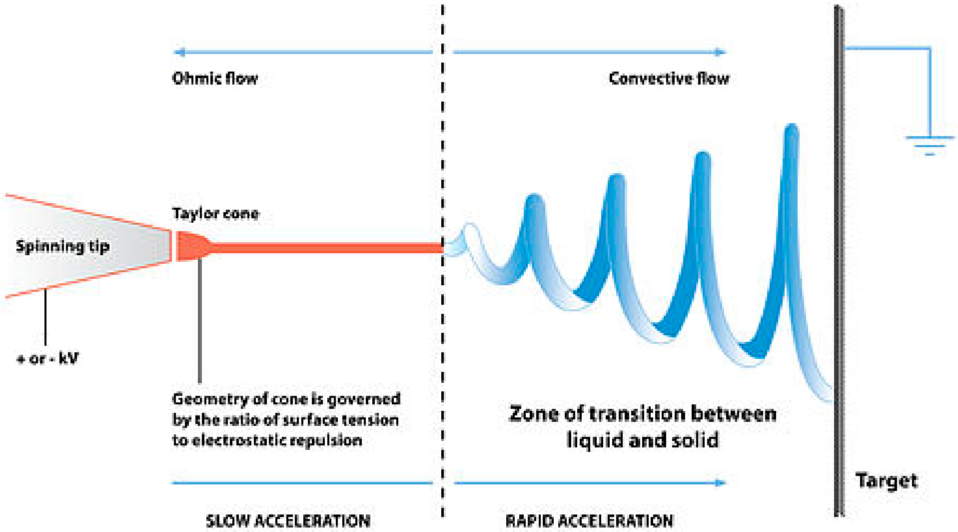
The Grimm 11-A (portable device) is an aerosol spectrometer used for extremely precise fine dust analyses within the size range 0.25 - 32 μm in 31 size channels and is useful for measuring particles inside in the model box, while the ppb RAE System “PGM-7240” is also a portable spectrometer which measures the volatile compounds at the exit of the model box.

GRIMM 11-A

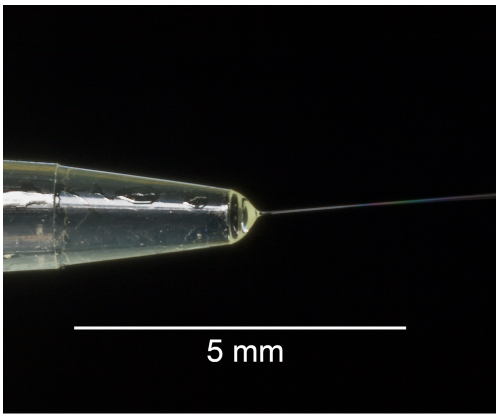
ppb Rae “PGM-7240”

1.2 ELECTROSPINNING PROCESS - HOW THE FILTER IS BUILT UP

Generally speaking, the electrospinning process is technically easy because we need a polymer in solution that has to be spinted into very little fibers thank to a high potential electric field.

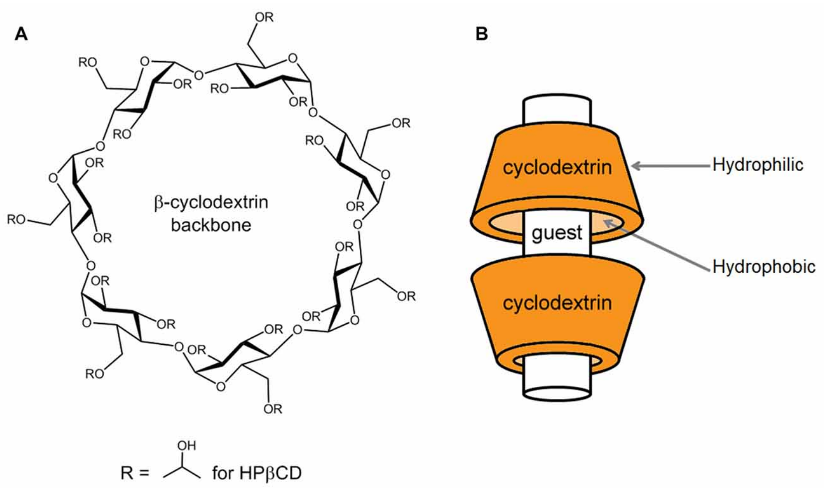


This method gives us the possibility to generate fibers with variable diameters (from micrometers to nanometers) and it is very convenient because it is necessary to have a syringe, a high tension survey and a collector. To start the process it is required an electric field which is stronger than the surface tension till the polymer begins creating the first jet called “Taylor’s cone”.



The famous “Taylor’s cone”

The support for the filter is positioned in the zone called “Target” and fibers start covering its surface. The distance between the spinning tip is very important because it conditions dimensions of fibers. The jet is linear in the first step of the process (we have a solution of polymers, so it is liquid); than we have the transition between liquid and solid because of the electric field and we have a jet that has a wiping motion: monitoring the speed of entire process, it is possible to obtain fibers of small diameters (near to nanometers). The best material for the electro spinning method is PLA (Polylactic Acid) and Cyclodextrins (CYD): these are compounds contained in filters we tested.



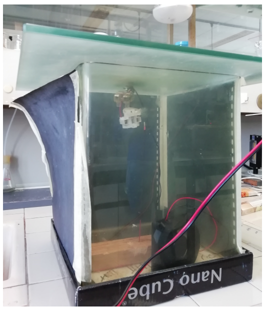
Cyclodextrin

PLA in rolls

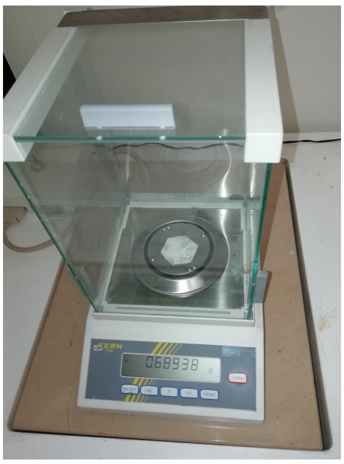
2. LABORATORY TESTING

2.1 WAY OF PROCEEDING AND OBJECTIVES OF THE RESEARCH

We did 3 tests for each filter using incense and measuring values with both instruments, one test with toluene (we used 2 µl and proceeded as previously described) which generates only VOCs (so we need only the ppb Rae) and one last test combining together incense and toluene.



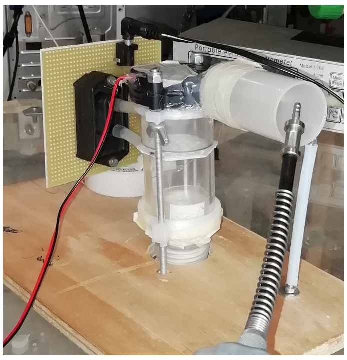
The first step for each day is to do an initial "blank test": proceeding in this way, it is possible to see how particles and volatile compounds interact with instruments without using the filter for an established quantity of time (like 20 minutes as an example), so this is what we really have in the model box (indoor air). Before this important test, we have to burn one stick of incense (in our case) inside another small and hermetic box with a fan that generates constantly particles and from what we have to take syringes to insert in the model box. Initially we let the fan working for around ten minutes, than we stop it and let bigger particles falling down into this chamber.



Scale

A filter used for our tests

After this, we can begin to test filters starting from inserting one syringe of 60 ml (or more if it is required by the situation) of incense into the model box in order to reach values bigger than 350 μm/m^3 PM (we can read this values looking at the Grimm instrument in the part reserved for particles bigger than 0.23 μm). When values go down to 300 (approximately), we can start inserting the filter chosen for the test and already weighted into the technical support and leave it working for 20 minutes (as we decided for our project).



During this time, it could be interesting to measure the wind speed and the pressure (because there is also a fan in the model box) using the Arduino program and kit connected to the laptop to read some values that give us the idea about how much the filter can hold back in terms of PM and VOCs. Talking about it, we can see how pressure decreases when we insert the filter and also the wind speed.

When the time has passed, we can remove the filter and weight it again to see if there are some differences in this term; then we can download datas from both instruments and start building graphs for each filter, also comparing them with other filters (made up with different compositions but tested in the same way). Last but not least, we can calculate the efficiency of filters using this formula:

$$E = (Co - Cn)/Co$$

E = Efficiency
Co = Initial concentration (maximum value in our case)
Cn = concentration after n-minutes (for example after 5 minutes it will be C5,…)

Speaking generally, it is possible to see a decrease of concentrations with parameters used in our tests but, for future studies, working with lower concentrations could be an hypothesis to observe if this tendency is confirmed.

The efficiency, otherwise, is not so relevant in our specific studies because we are not in a complete plug-flow process. In that case, measuring it could be so interesting to define in a better way how a filter works but in our case it is mentioned just to give a more defined background about processes connected to air pollution removal.

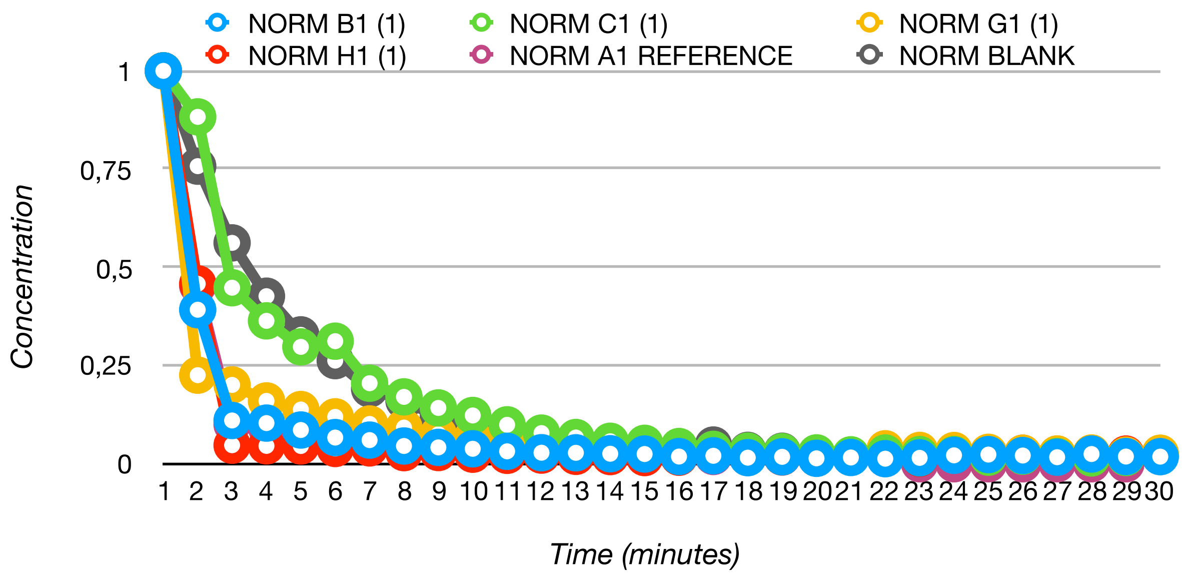
3. DATA TREATMENT AND DISCUSSION OF RESULT

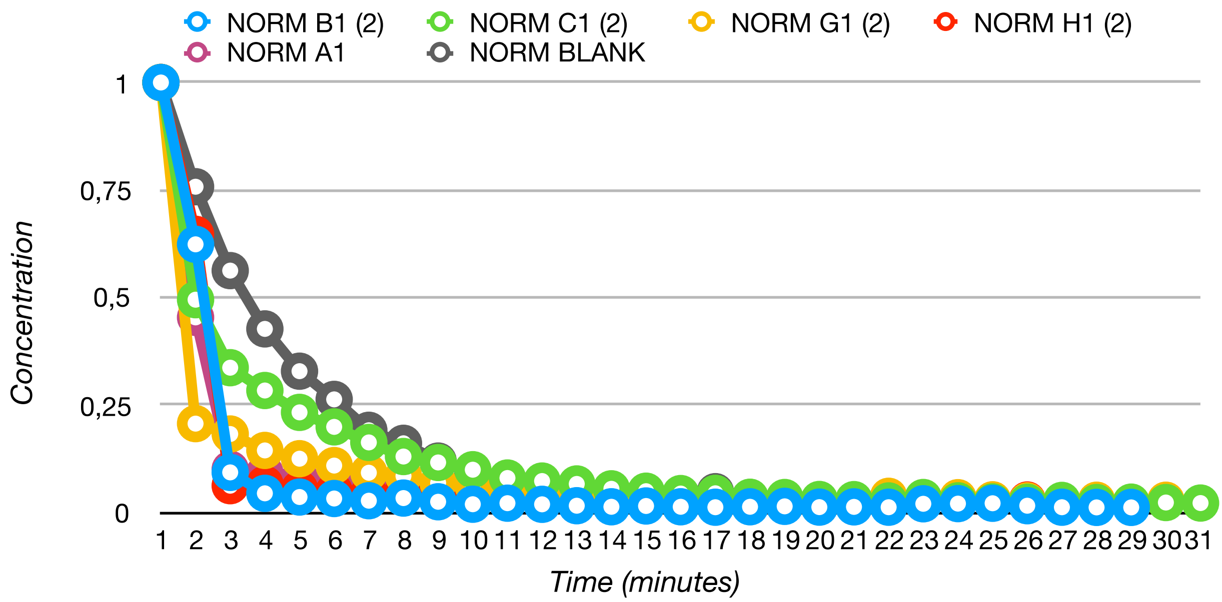
Here there are some graphs which are representative for the behavior of filters tested as previously described.

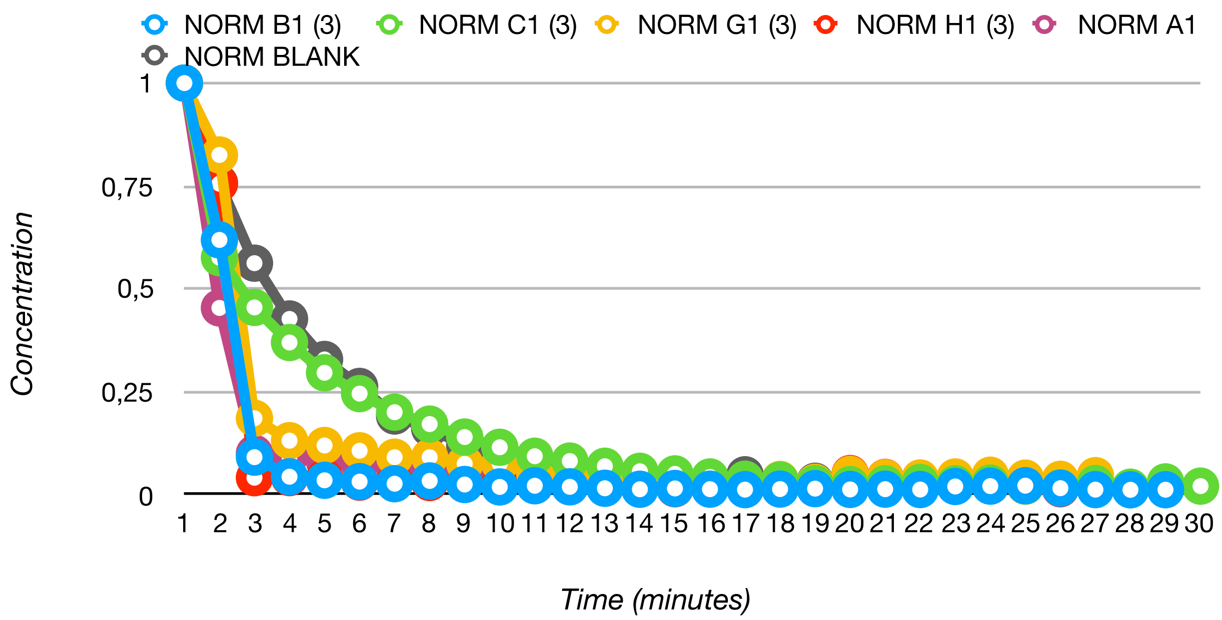
All graphs were made with the program “Numbers” elaborating datas every day after each test to catch immediately something good or bad out of the test results. Values downloaded from the GRIMM and from the ppb RAE system are the basis for what is possible to see downside.

Graphs are all in a “normalized” scale in order to give a starting point equalized for all filters tested. Moreover, ***the X-Axis show details about time (expressed in minutes), while the Y-Axis works in a range from 0 to 1 for the concentration*** (which is normalized as we said previously). Proceeding in this way, it is possible to see how filters work and this is also useful to compare them better (without starting from different points). The normalized value is substantially the highest value divided for itself so that the starting point is 1 and the others are less than 1 because we also divide them for the highest. These are results:

- Final graph for test with incense - Filters A1(reference), B1, C1, G1 and H1 + the initial blank test (1°, 2° and 3° test in sequence) —> values are normalized with maximum of each test

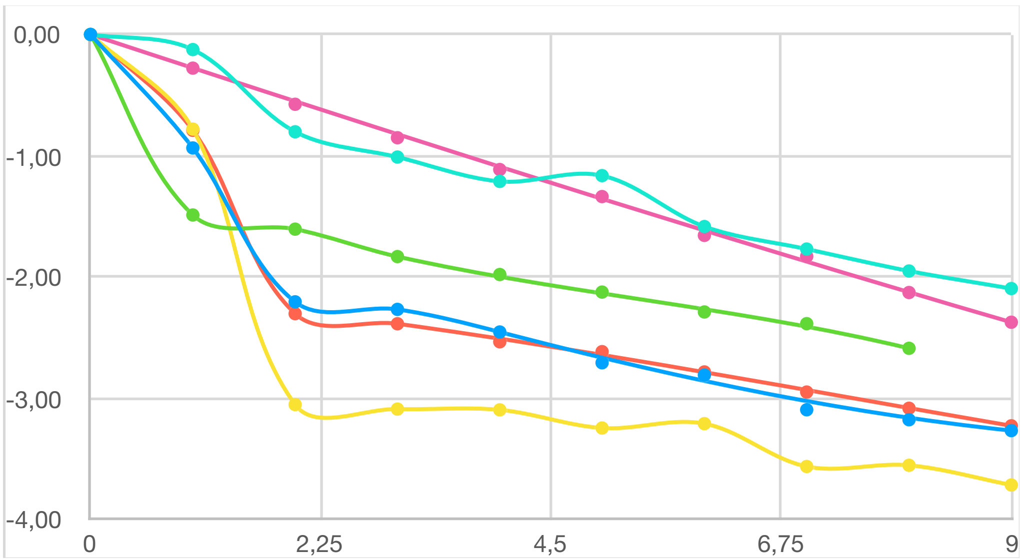

NORM B1 (1)
NORM C1 (1)
NORM G1 (1)
NORM H1 (1)
NORM A1 REFERENCE
NORM BLANK
Concentration
1
0,75
0,5
0,25
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30
Time (minutes)


NORM B1 (2)
NORM C1 (2)
NORM G1 (2)
NORM H1 (2)
NORM A1
NORM BLANK
Concentration
1
0,75
0,5
0,25
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31
Time (minutes)

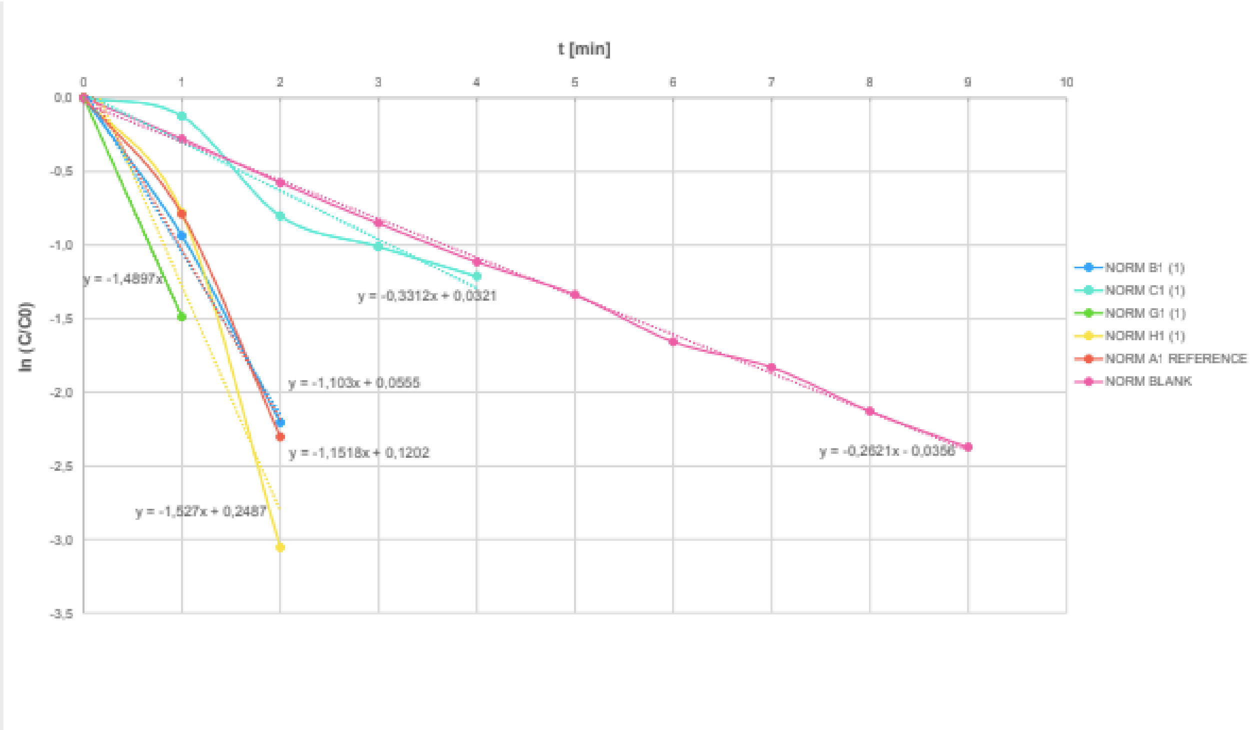

NORM B1 (3)
NORM C1 (3)
NORM G1 (3)
NORM H1 (3)
NORM A1
NORM BLANK
Concentration
1
0,75
0,5
0,25
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30
Time (minutes)

Having a general look to these three graphs, it is possible to observe that the tendency is quite constant: values decrease rapidly within 3-5 minutes and the graph (for each filter) could be assimilated to a line (the entire graph as a parabolic trajectory but after a maximum of 10 minutes it decrease constantly like a line). These filters "of the first column" (as we called them in our tests to recognize their specific composition different from the others) were tested with incense which generates high quantities of particles (the famous air pollution we were studying). Looking at how the line decrease to very low values (so close to 0), it is possible to have an idea about the removal efficiency of the filter. This is the most interesting thing to extrapolate from the upside work.

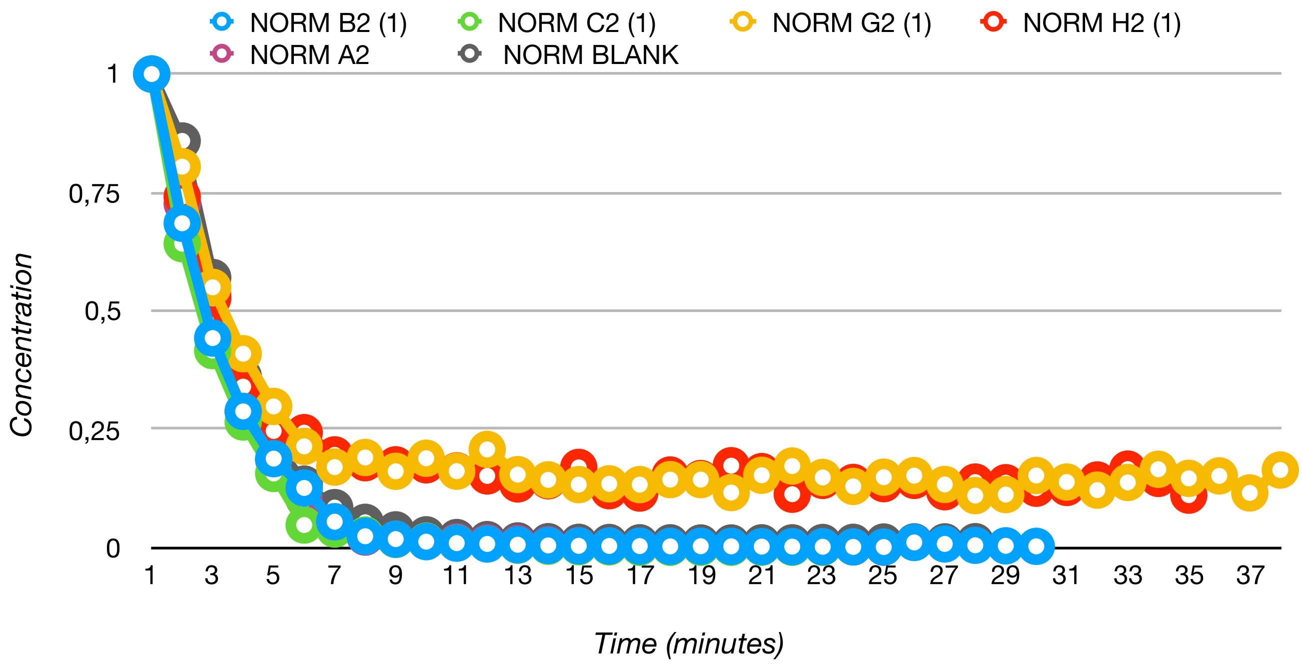
In addition to what said previously, it is possible to see another useful thing in graphs I will put downside: the decay lines which are normalized to the *ln(C/Co)* to estimate the decreasing speed of VOCs with the flux. We did this for not longer times (maximum 9 minutes) but the tendency of the graph is quite constant. This is only an example that could help the research to have a new parameter to measure.

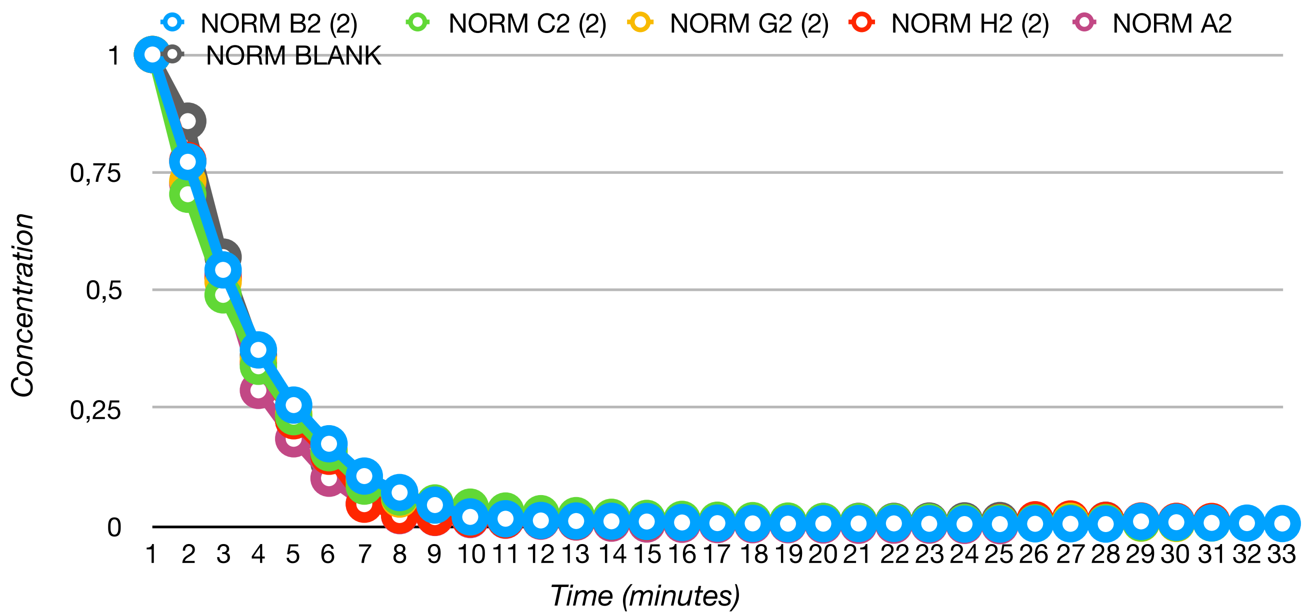


Graph normalized to *ln(C/Co)*

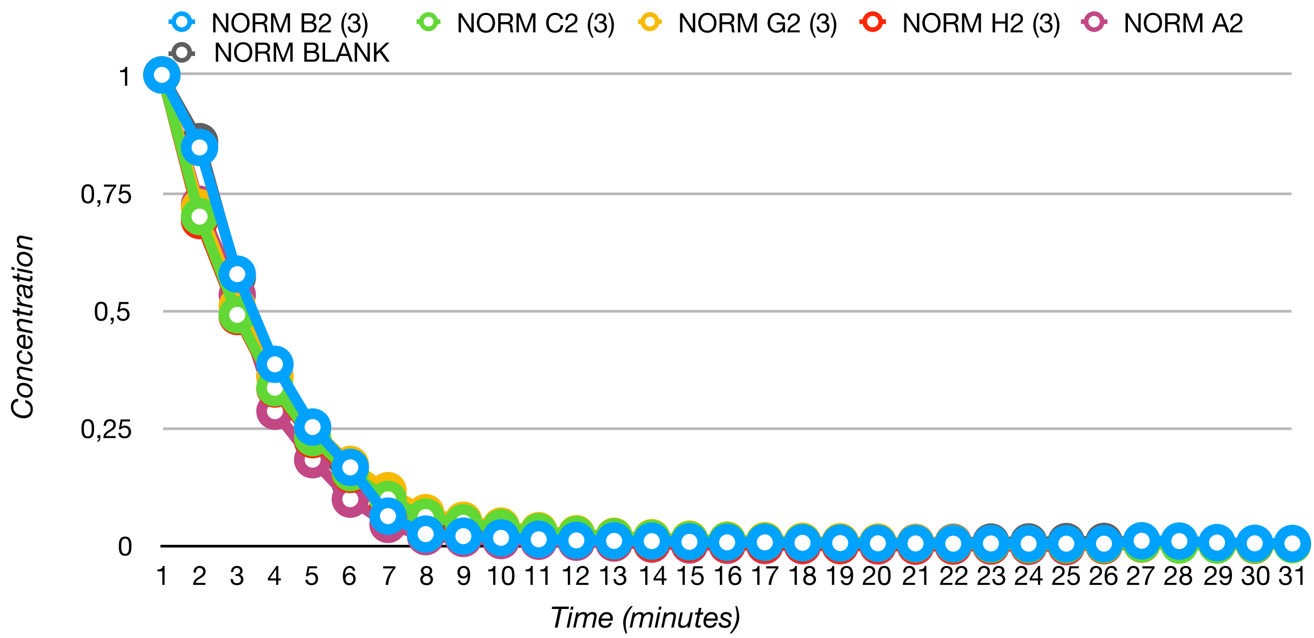


- Final graph for test with toluene - Filters A2, B2, C2, G2 and H2 + the initial blank test (1°, 2° and 3° test in sequence) –> values are normalized with maximum of each test





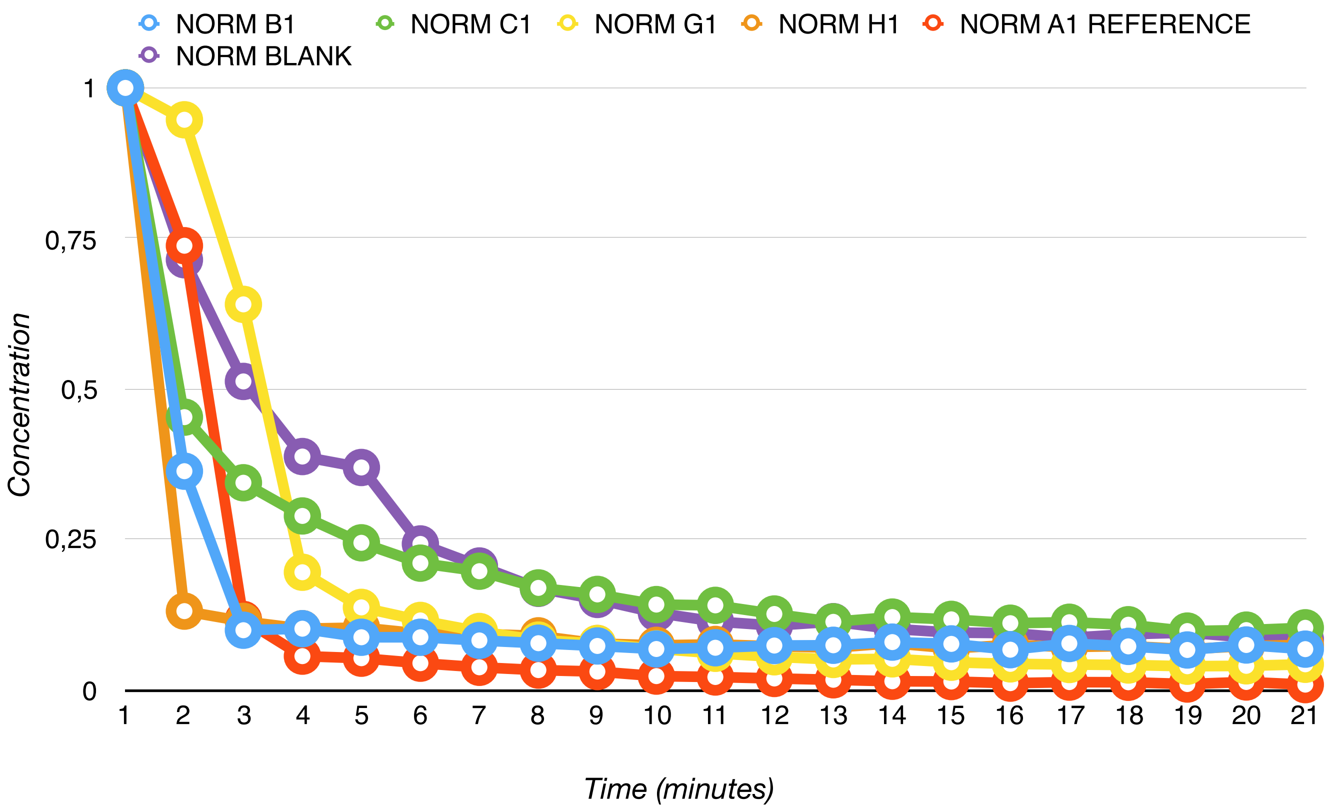
NORM B2 (2)
NORM C2 (2)
NORM G2 (2)
NORM H2 (2)
NORM A2
NORM BLANK
Concentration
1
0,75
0,5
0,25
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33
Time (minutes)



NORM B2 (3)
NORM C2 (3)
NORM G2 (3)
NORM H2 (3)
NORM A2
NORM BLANK
Concentration
1
0,75
0,5
0,25
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31
Time (minutes)

In these graphs made up with filters “of the second column” tested only with toluene, it is possible to see a tendency which is quite similar for all filters (despite the fact that they present different characterizations). We could only mention the first graph where it is clear that two filters (G2 and H2 - yellow and red lines) have a kind of strange behavior comparing them to the others. We ended up with the conclusion that this could depend on something occurred to instruments at the moment of tests (only on this day as it is possible to observe having a general view).

- Final graph for test with incense and toluene combined together in the model box - Filters A1, B1, C1, G1 and H1 + the initial blank test –> values are normalized with maximum of each test



This graph shows results of test made up with incense and toluene combined together for filters “of the first column”. The tendency is quite constant and follows a parabolic trajectory. In these tests we also tried to measure the decreasing speed of removal efficiency in time. The result (for example looking at filter B1) is the black line: measuring the pendency of this geometric element, it is possible to obtain the value of the removal efficiency for the first six minutes (we can do it for longer or shorter times without problems) for the specific filter.

To complete the analysis of filters and give a better background to the discussion of results, we measure the wind speed and the pressure at the exit of the box model with the Arduino’s software linked to the laptop by a usb cable connected to a small electronic device which can show us lots of datas about these features (we took an average). It is possible to see results of this work looking at the table downside. We also inserted the weight of filters before and after the test in order to measure some variations connected to the removal efficiency.

Filter		Windspeed (in m/s)			Mass of the filter BEFORE (g)	Mass of the filter AFTER (g)	Mass after measurement %
		centre	right	left			
A1 *Incense*		3,20	3,60	3,86	1,23934	1,23948	99,989
A2		2,34	2,25	2,76	1,23560	1,23566	100,005
B1 (1) *Incense*	I	1,78/2,08	1,29/1,54	1,71/2,01	1,27113	1,27108	99,996
	II	1,44/1,40	1,99/1,83	1,23			
B1 (2) *Incense*	I	1,62/1,81	1,71/1,40	1,89/1,93	1,27099	1,27100	100,001
	II	1,44/1,59	1,11/1,33	1,71/1,96			

4. MORPHOLOGY CHARACTERIZATION

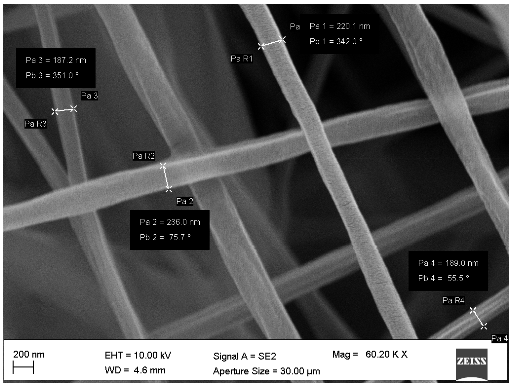
All filters tested were made up with the "electrospinning" method and they are composed in this way:

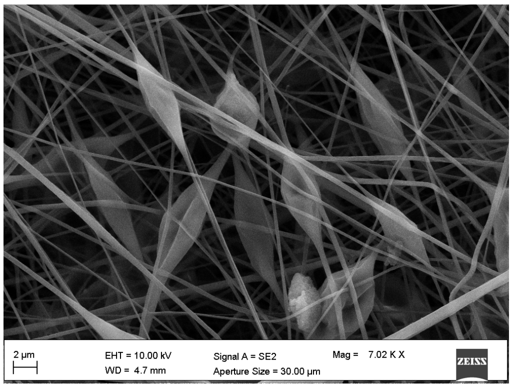
FILTER A –> 0,08 g/ml PLA; DCM-DMF –> 80:20; 19,5 kV; 1,5 ml/h; 13 cm;
FILTER B –> PLA + 15% Cyclodextrins
FILTER C –> PLA + particles of Cyclodextrins; 0,8 mg/ml DCM-DMF–> 9 cm and 19 kV; 3,42 g/10 ml pure DMF –> 12 cm and 21 kV
FILTER G –> PLA + 1,5% of Cyclodextrins in solution; 1,5 ml/h; 19 kV; 6-9 cm;
FILTER H –> PLA + Cyclodextrins; 0,08 mg/ml per 1 h –> 9 cm and 19 kV; 0,342 mg/ml per 30 minutes –> 12 cm and 22 kV

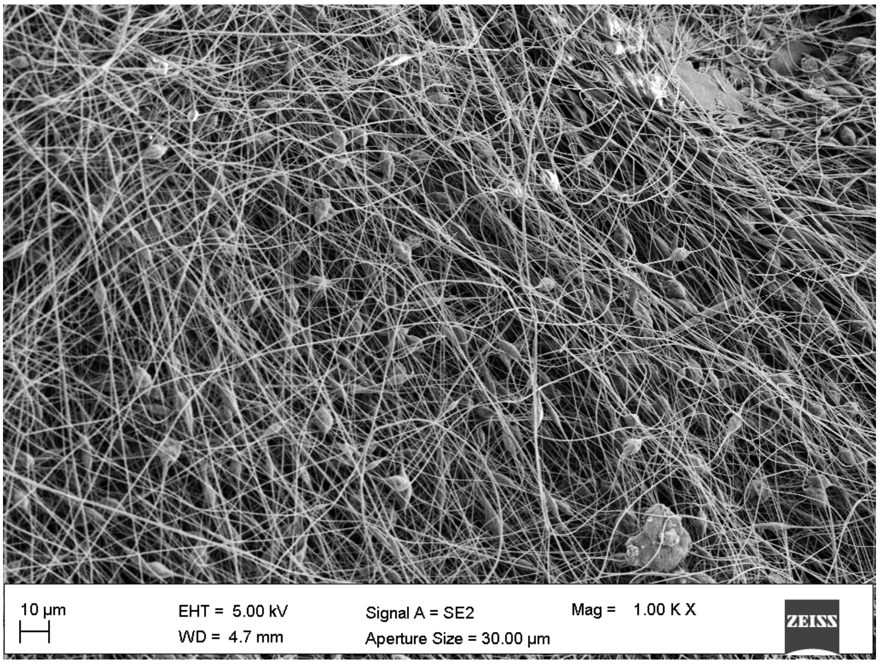
These datas explain compositions of filters in a detailed way and also other two important things: the voltage and the distance between the syringe and the support while making the filter. This description made up with numbers is important to give a background of what we are testing also to predict in a way how the filter could work (generally speaking).

After tests with incense and toluene and after building graphs, we had the opportunity to see the micro-structure of each filter using the SEM (Scanning Electron Microscope): here there are some images which explain the internal composition of filters.

FILTER A

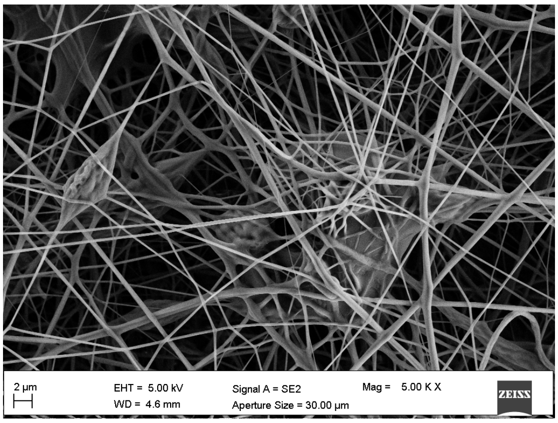


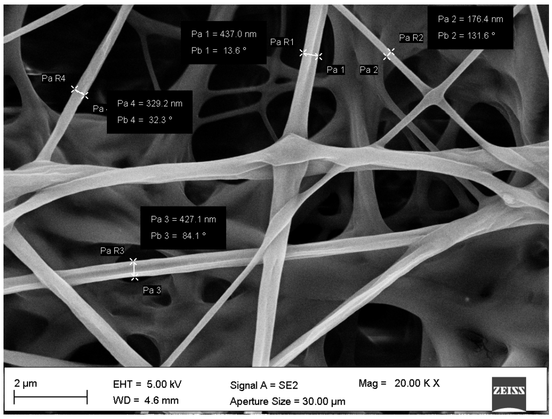


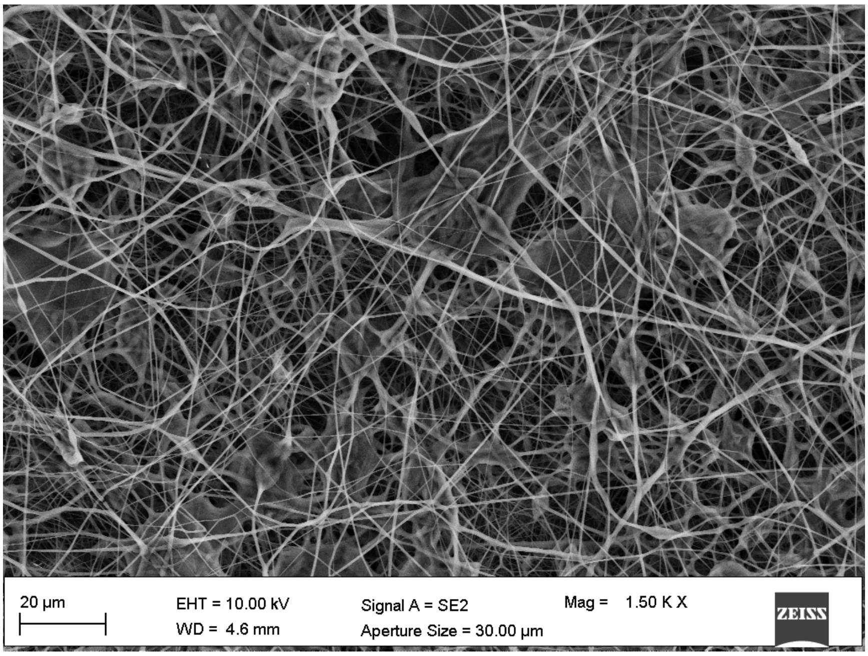


Having a look to these photographs, it is possible to distinguish immediately fibers which composes the sample “A”. Dimensions are around 226,6 nm (taking an average of some values).

FILTER B

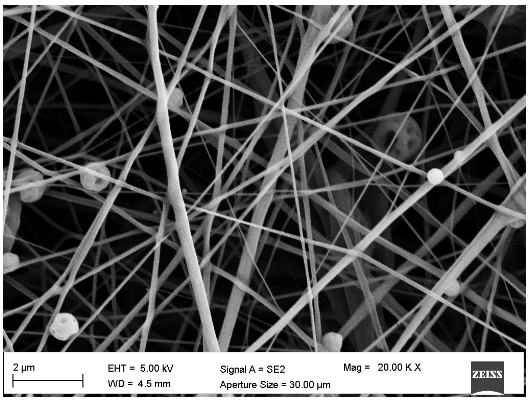


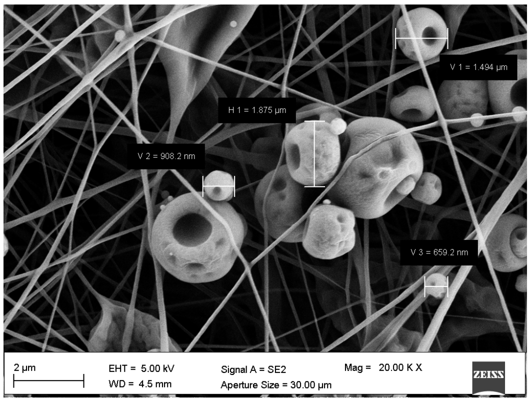


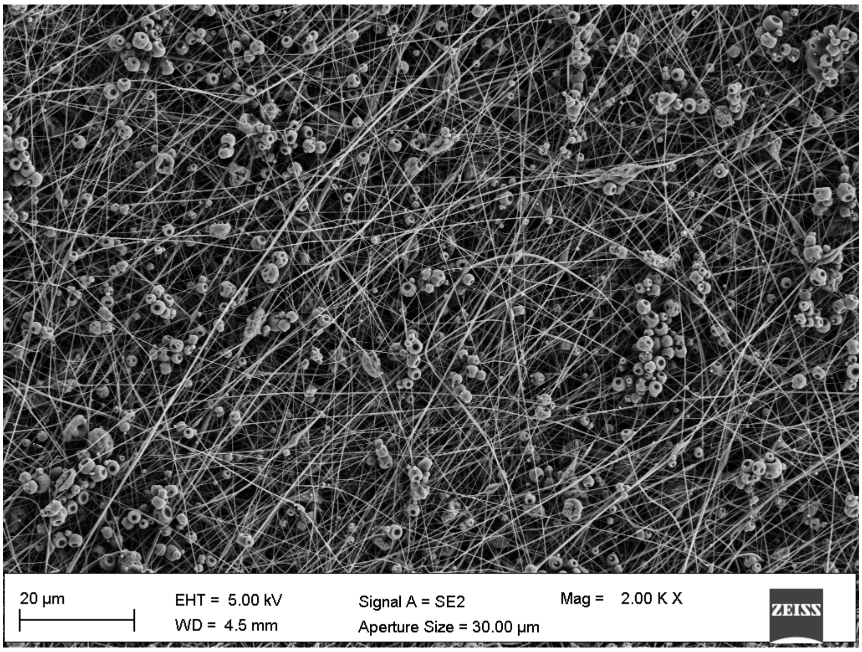


Scanning the sample "B" it is possible to observe a structure made up with fibers linked together to create a complex net. Dimensions are around 342,4 nm.

FILTER C

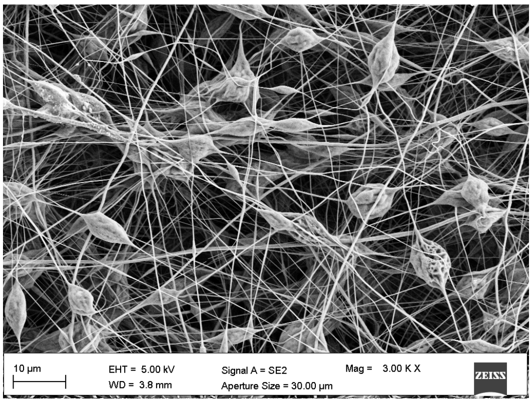


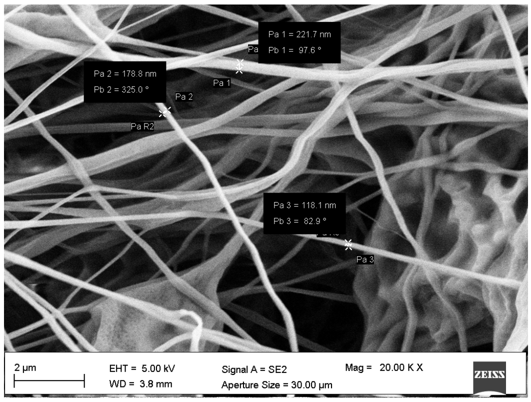


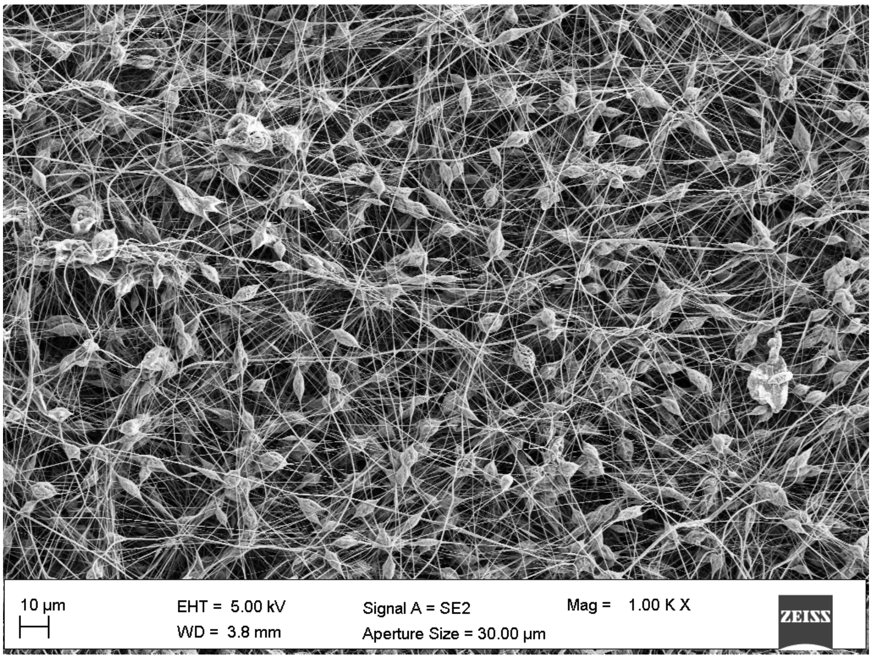


Having a look to these photographs, we can see a structure a little bit different from samples “A” and “B”: in fact, images of sample “C” show a net of fibers in which are mixed cyclodextrins (the micro “balls” shown at the top). Dimensions are around 114,1 nm for fibers and around 1196,6 nm for cyclodextrins particles.

FILTER G

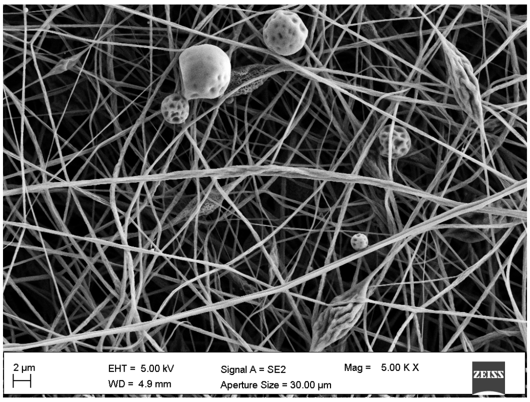


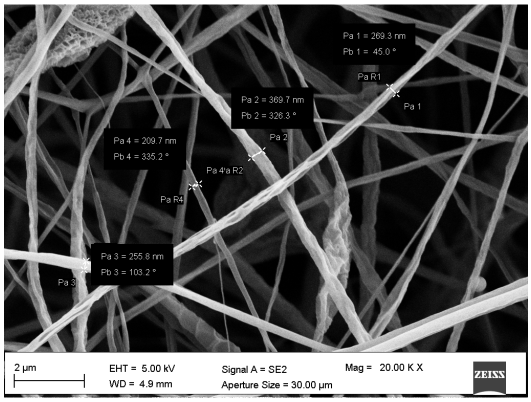


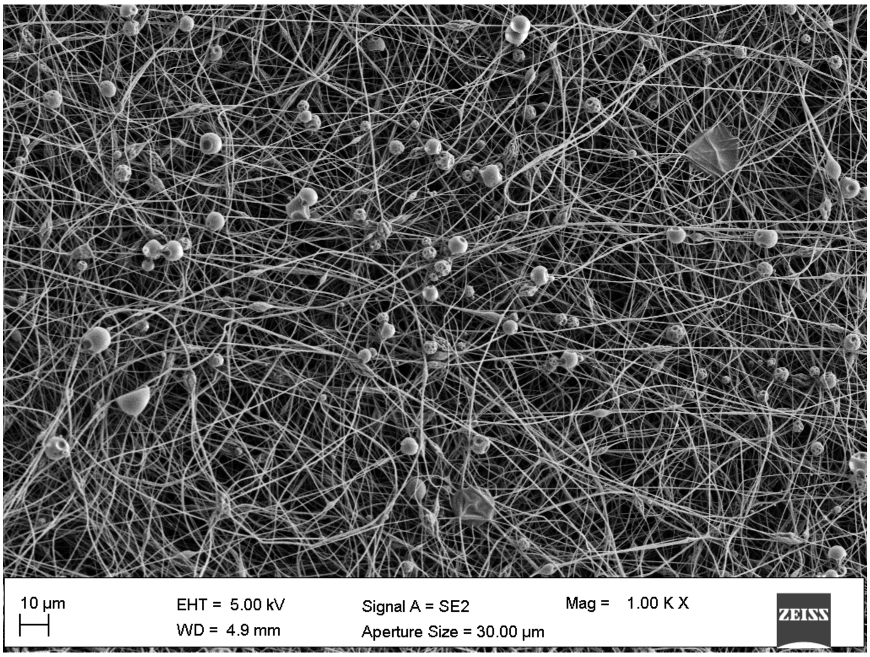


The sample "G" shows another kind of structure which is so similar to yarn. PLA and cyclodextrins sprayed in a solution create this particular grid, a little bit confusing in front of samples described below. Dimensions are around 172,9 nm (always taking an average of some values measured with the SEM).

FILTER H







Having a look to photographs of sample "H", it is possible to observe a structure very close to the "C" one because of the famous net of fibers in which are embedded cyclodextrins. Dimensions are 252,6 nm.

Scanning all filters with the SEM microscope, it is interesting to see dimensions of fibers and particles of cyclodextrins (as shown in some photographs before) and measure them to give a background for the removal efficiency of each filter.

The SEM microscope in our University

The SEM microscope works using series of electromagnetic coils that pull the beam back and forth, scanning it slowly and systematically across the specimen's surface. Instead of traveling through the specimen, the electron beam effectively bounces straight off it. The electrons that are reflected off the specimen (known as secondary electrons) are directed at a screen where they create a TV-like picture. This “laptop” produces high-quality 3D images sending them directly from the microscope to the screen. The quality of photograms depends on how the support has been made and also on the experience of who moves lens upside the surface of each specimen.

To prepare a specimen, it is required to cut a small piece of paper with the composition of each filter (this paper is sticky and it is the same used to prepare filters so it maintains exactly properties, because during the electrospinning method it is behind so it catches particles and fibers) and put it up to a support (with graphite or gold) that has to be insert in the microscope. Every support contains only one piece of paper so it is very easy to have an order in the microscope when the scanning process is on.

Taking the average of all measurements made up with the SEM microscope, these are the results:

FILTER A: dim(A) = 226,6 nm (only fibers)

FILTER B: dim(B) = 342,4 nm (only fibers)

FILTER C: dim(C - cyclodextrins)= 1196,6 nm ; dim (C - fibers)= 113,1 nm

FILTER G: dim(G) = 172,9 nm (only fibers)

FILTER H: dim(H - cyclodextrins)= 3476,3 nm ; dim (H - fibers)= 252,5 nm

Another interesting thing to mention is that, measuring fibers of filters, these results have an important influence speaking about efficiency because, as it is possible to see in graphs upside, bigger fibers considerably increase removal efficiency (graphs require a bigger scale to show some variations in this way) while smaller fibers reduce analyzed particles in the model box a little bit less. Making trials with longer times and more incense or toluene surely show better results in this term.

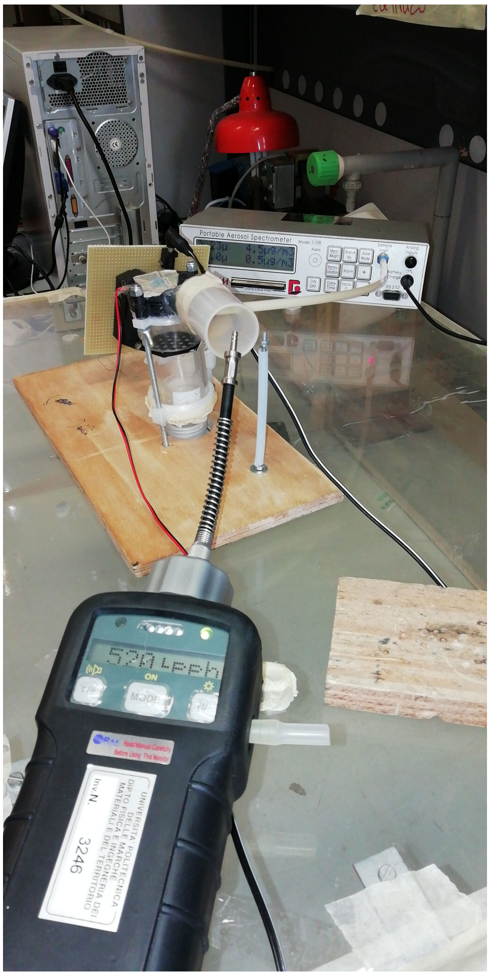
5. CONCLUSIONS

At the end of this analysis conducted in the thesis, it is possible to affirm that new technologies about air pollution removal are becoming better and better through years, especially working on nano dimensions which is the best scale for treating this kind of problem. What we did in our laboratory at the university is just giving a background about how the entire process works also providing some result obtained. Anyway, our way of proceeding is not the only one that shows significant results because, as an example, if you work on variables such as time of exposition, increased speed of the fan inside the model box and having a recirculation and not an eject of the flux, it is possible to show better (in graphs especially) the behavior of each filter in front of particles and VOCs. We fixed our variables as illustrated before but the entire process could show other results also inserting a large quantity of incense (and not only one syringe of 60 ml) and toluene.

So this must not be considered the only truth or the perfect conclusion of the work but it is a kind of "open conclusion": working of some little details you can obtain another better background of filters and how they remove pollution. It is possible to vary many things and find the "perfect mix" at the moment for what you can reach with your studies.

As an advice, firstly It is possible to increase time of exposition from 25 to 60 minutes eventually recharging incense or toluene and let the filter work for this time checking if the behavior could be similar. This could be extremely helpful to verify in how much time the filter is being saturated and, as a consequence, the specific durability because each filter has a different "way of catching" particles and VOCs.

Another possibility linked to the previous one is to close the exit from the model box and generate a flux which recirculates inside it. These are only two of lots of variables to change to study better the filter itself.



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