Qualitative and Quantitative analysis of some imported cigarette samples locally used in Saudi Arabia by Scanning Electron Microscope and Energy Dispersive Spectroscopy

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Abstract: Seventeen samples of imported cigarette locally used in Saudi Arabia were investigated for qualitative and quantitative constituents. Qualitative and quantitative analysis were done using Scanning Electron Microscope and Energy Dispersive Spectroscopy (SEM-EDS). Qualitative analysis shows that the major elements presented in the samples are Carbon and Oxygen, followed by Potassium, Calcium, Chlorine, Magnesium, Silicon, Aluminum, Sulfur and Phosphorus. Samples (S10), (S17) present a significant amount of Carbon and low amount of Oxygen. Magnesium ranged from 0.44 (S12) to 1.28 (S10). Aluminum ranged from 0.06 (S9) to 0.11 (S12). Silicon ranged from 0.10 (S3) to 0.90 (S10, S14). Phosphor ranged from 0.09 (S1) to 0.28 (S14). Sulfur ranged from 0.11 (S1) to 0.65 (S14). Chlorine ranged from 0.37(S16) to 5.25 (S8) and calcium ranged from 1.19 (S12) to 6.07 (S10). It is clear that sample S10 high in Si, K, Ca with respect to other brands of cigarettes. While, sample S14 high in Si, P, S with respect to other brands of cigarettes. The indicative ratio O/C for Black Carbon (BC) ranged from 0.1 for sample S10 and S17 to 0.81for sample S11.

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Introduction:

1. Cigarettes and Smokers :

It is well known that cigarette Tobacco is very harmful to health because of its contents. According to WHO, at least one person dies per 10 second as a result of cigarette smoking (WHO **1999,** Environmental Health Criteria, 211). Tobacco is a sensitive plant prone to many diseases. It therefore requires high chemical inputs up to sixteen applications of pesticide are recommended during one three-month growing period. Some of the chemicals are absorbed by the plant and residues remain in the final tobacco product. Residues of some pesticides used to grow tobacco remain on the tobacco leaf and can be present in cigarettes (Otmar Geiss *et al., 2007*). So,most of the constituents originate from the tobacco leaf while some arise from growing conditions such as soil, atmosphere and agricultural chemicals used (Yebpella *et al.,*2013). Cigarette ingredients mentioned on a cigarette pack include nicotine, tar, carbon monoxide and additives. Nicotine is inhaled into the lungs and reaches your brain in just six seconds. Addiction to nicotine poses very serious health risks in the long run. Nicotine in small doses acts as a stimulant to the brain. In large doses, it's a depressant, inhibiting the flow of signals between nerve cells. In even larger doses, it's a lethal poison, affecting the heart, blood vessels, and hormones. Nicotine in the bloodstream acts to make the smoker feel calm. As a cigarette is smoked, the amount of tar inhaled into the lungs increases, and the last puff contains more than twice as much tar as the first puff. Tar is a mixture of substances that together form a sticky mass in the lungs (http://www.quitsmokingsupport.com/whatsinit.htm). The lungs of smokers collect an annual deposit of one-quarter to one and one-half pounds of the gooey black material. Carbon monoxide (CO) is a poisonous, colorless and odorless gas that is produced as a result of incomplete burning of carbon-containing fuels. Cigarette smoke can contain high levels of CO. When inhaled, CO quickly binds with hemoglobin in red blood cells in the lungs. This can affect the amount of hemoglobin available for the transport of oxygen throughout the body, which may in turn lead to symptoms of CO poisoning. That means a victim of carbon monoxide poisoning has less oxygen getting to important organs like the heart and brain **(Foulds** *et al.*, 2008). Smoke chemicals pollute the enclosed air spaces of homes, offices, conference rooms, restaurants, and environments. Every time someone lights up a cigarette, cigar or pipe, tobacco smoke enters the air from two sources. The first is mainstream smoke, which the smoker pulls through the mouthpiece when inhaling or puffing. Non-smokers are also exposed to mainstream smoke after it is exhaled. The second, and even more dangerous source, is sidestream smoke, which goes directly into the air from the burning tobacco. Exposure to secondhand smoke (environmental tobacco smoke, passive smoking) has been shown to pose a similar health risk as that caused by direct smoking (Salman *et al*., 2012). Health authorities have long considered that lung cancer risks due to cigarette smoke exposure were related to the presence of tar (Otmar *et al., 2007*). Increased risks for cardiovascular disease in general and coronary artery disease in particular have been documented for active and second- hand smoke as well as ambient particulate matter (Annette Peters, 2009). Also, black carbon is produced from incomplete combustion of a biomass, as a residues of tobacco smoke. Using human and animal exposure data, researchers can estimate the amount of black carbon deposited into lungs under a variety of breathing conditions. Such research has contributed to global awareness of black carbon's health effects.

2. Scanning Electron Microscope and Energy Dispersive Spectroscopy :

It is important to determine cigarette contents with accurate techniques such as Scanning Electron Microscope and Energy Dispersive Spectroscopy (SEM-EDS). All elements from atomic number 4 (Be) to 92(U) can be detected in principle, though not all instruments are equipped for 'light' elements (Z< 10). Qualitative analysis involves the identification of the specific lines in the X-ray spectrum and is fairly straightforward owing to the simplicity of X-ray spectra. Quantitative analysis (determination of the concentrations of the elements present) entails measuring line intensities for each element in the sample and for the same elements in calibration Standards of known composition (Sameh *et al., 201*3). The basic design of a modern SEM includes an electron source, one or more sets of condenser lenses, an objective lens, and several detectors. Electrons beam generated are focused to an adjustable spot size utilizing electromagnetic condensing lenses. After condensing the beam to the desired spot size, the objective lens directs the beam to raster the sample surface providing illumination of the sample. Upon the beam striking the sample, electrons are released in the form of secondary electrons from the ionization of the surface or in the form of backscattered electrons from elastic beam-surface interactions. The secondary electrons and backscattered electrons are collected by two separate detectors. Additionally, the removal of inner shell electrons from atoms in the sample causes a release of energy in the form of an X-ray of a characteristic wavelength, which may be detected and quantified by the EDS detector[2]. To prepare samples for SEM-EDS analysis, the surface must be relatively conductive to avoid the buildup of excess negative charge due to the high beam current utilized in SEM. Typical beam currents for SEM range from 0.2-40 keV allowing magnifications of up to 500,000 times with a spot size ranging from 0.4-5 nm. In order to increase the conductivity of non-conductive samples, many samples receive a 10-30 nm coating of carbon particles deposited through a sputter coating instrument. Samples are introduced to the SEM through a series of vacuum lock chambers allowing a gradual reduction in pressure surrounding the sample. This is necessary to pass the sample into the vacuum environment accessible by the electron beam. Once the sample is positioned within the SEM stage, the electronic beam is switched on and the secondary electron detector is activated allowing for topographical imaging of the sample. Secondary electrons are detected using an Everhart-Thornley detector which collects the negative secondary electrons emitted from samples using a collector at a high positive potential. Once the secondary electrons strike the collector, photons of light are released and enter a photomultiplier tube where the photons are amplified as electrons for image processing by the software. The sample stage moves either manually or through computer control allowing imaging of the entire sample surface. Once operational, the backscattered electron detector may be switched on providing contrast in the image based on the average atomic number of elements within the sample bulk. Backscattered electrons are detected through use of a Robinson or semiconductor detector placed directly above the sample to capture the high energy backscattered electrons from the sample (Dalby *et al.,* 2010).

2. Experimental Method and Measurements:

***1*. Samples and samples preparation:**

Fifteen different samples of Cigarette has been collected from local market. Two more samples are pipe tobacco. All samples are foreign brands imported to Saudi Arabia. All samples were grinded to 200 mesh and sieved in order to increase homogeneity for analysis by SEM-EDX analysis, small quantities with reasonable size and shapes were taken to get representative specimen which should not exceed holder diameter. The specimen was fixed on a carbon tape pasted on the holder using spatula.

2. Measurements:

Seventeen different samples of Cigarette, locally used in Saudi Arabia, were investigated using Scanning Electron Microscope and Energy Dispersive Spectroscopy (SEM-EDS). SEM utilizes a powerful beam of electrons directed at the sample allowing for observation of spatial features of sample at increased depths. Meanwhile, the energy dispersive X-ray (EDS) system determines the elemental composition of samples based on the emission of characteristic X-ray wavelengths arising from the interaction of electrons with atoms in the sample surface.

The SEM used in this study is a JEOL JSM-6510LV model with resolution of 1pA0–11A0 (Scanning Electron Microscope (SEM) and Energy Dispersive X-ray (EDX) manual, Jeol) Scanning

Electron Microscope (SEM) and Energy Dispersive X-ray (EDX) spectrometer is employed for the measurement. The specimen was placed inside the holder and it was fixed inside the instrument at working distance 10 mm (WD10 mm), voltage = 30 kV, magnification value = 100 and spot size = 50nm. Quant method was used for analysis. Likewise, the EDS detector can run simultaneously to provide detailed information about the elemental composition of sample in the form of an X-ray spectrum. A silicon drift solid state detector is used because of its better energy resolution, the detector giving output pulses proportional in height to the X-ray photon energy is used in conjunction with a pulse height analyzer (in this case a multichannel type). Incident X-ray photons cause ionization in the detector, producing an electrical charge, which is amplified by a sensitive preamplifier located close to the detector. Both detector and preamplifier are cooled with liquid nitrogen to minimize electronic noise. Due to the quantized nature of atomic shells, the energy of the X-ray for each element has a characteristic energy used for identification of elements present in a sample. Black carbon is produced from incomplete combustion of a biomass. For identification of black carbon (BC), it has been recommended to monitor atomic O/C ratios using SEM/EDX techniques. This requires that O/C ratios can be accurately determined. Indicative for BC is the low O/C ratios (O/C=0.2–0.60) (Sonja *et al.*, 2005), (Hedges *et al.*,2000).

3. Results and Discussion:

For qualitative analysis, by finding the elements present in the samples and to obtain an estimation of how much of each is present in the sample. This can be done by identifying the peaks in the X-ray spectrum. Figure (1) shows the SEM image of sample S1 and Figure (2) gives the x-ray spectrum for the sample S1.

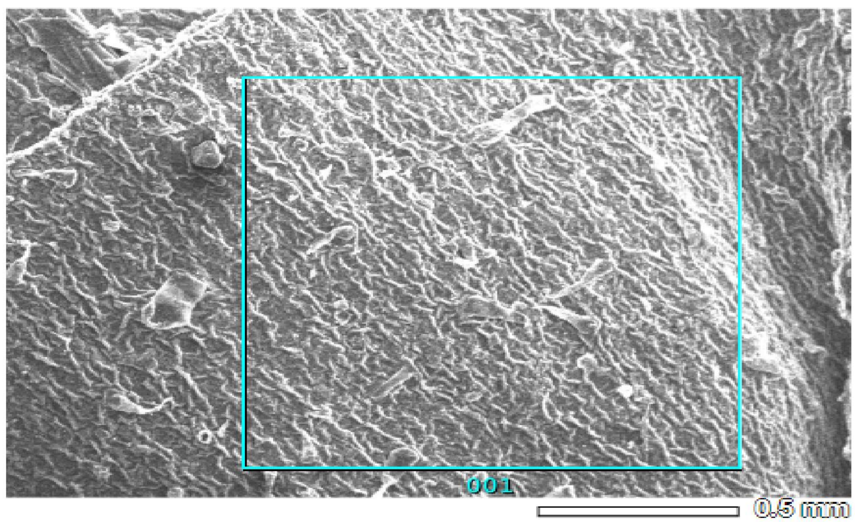


Figure 1. shows the SEM image of sample S1

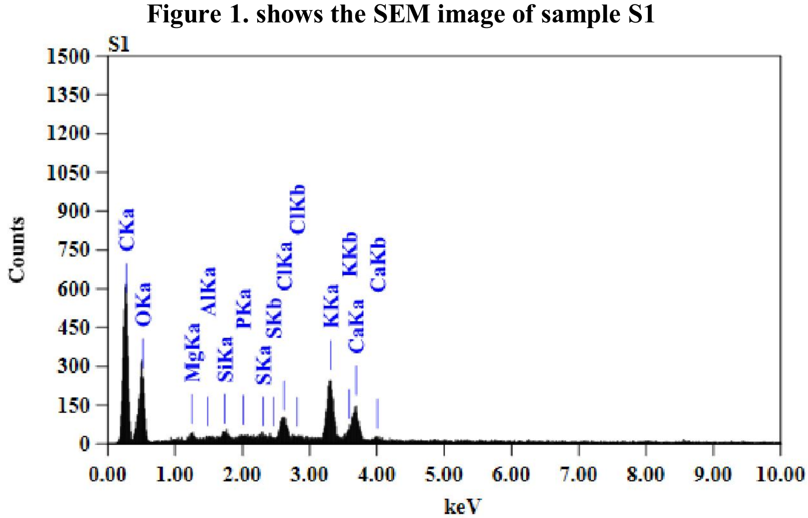


Figure 2 gives the X-ray spectrum for the sample S1

Results of qualitative analysis using SEM-EDS showed that there are ten elements:

1. ***Carbon***: elemental carbon is of very low toxicity. Health hazard data presented here is based on exposures to **black carbon**, not elemental carbon. Chronic inhalation exposure to black carbon may result in temporary or permanent damage to lungs and heart.

2. ***Oxygen***: every human being needs oxygen to breathe, but as in so many cases too much is not good. If one is exposed to large amounts of oxygen for a long time, lung damage can occur. Breathing 50-100% oxygen at normal pressure over a prolonged period causes lung damage.

3. ***Magnesium***: inhalation of magnesium dust may irritate mucous membranes or upper respiratory tract. Viewing of burning magnesium powder without fire glasses may result in "Welder's flash", due to intense white flame. Skin: embedding of particle in skin. Magnesium oxide fume is a by-product of burning magnesium.

4. ***Aluminum***: inhalation of finely divided aluminum and aluminum oxide powder has been reported as a cause of pulmonary fibrosis and lung damage. This effect, know as Shaver's Disease, is complicated by the presence in the inhaled air of silica and oxides of iron. May also be implicated in Alzheimer's disease.

5. ***Silicon***: crystalline silica (silicon dioxide) is a potent respiratory hazard. Silicon crystalline irritates the skin and eyes on contact. Inhalation will cause irritation to the lungs and mucus membrane. Irritation to the eyes will cause watering and redness. Reddening, scaling, and itching are characteristics of skin inflammation. Lung cancer is associated with occupational exposures to crystalline silica specifically quartz and cristobalite. Exposure to breathable crystalline silica is associated with bronchitis, chronic obstructive pulmonary disease (COPD) and emphysema.

6. ***Phosphorus*** in its pure form has a white color. White phosphorus is the most dangerous form of phosphorus that is known to us. When white phosphorus occurs in nature this can be a serious danger to our health. White phosphorus is extremely poisonous and in many cases exposure to it will be fatal.

7. ***Sulphur:*** globally sulphuric substances can have the following effects on human health (http://www.lenntech.com/periodic/elements/o.htm#ixzz3BjEmD8ae).

- Neurological effects and behavioural changes
- Disturbance of blood circulation
- Heart damage
- Effects on eyes and eyesight
- Reproductive failure
- Damage to immune systems
- Stomach and gastrointestinal disorder
- Damage to liver and kidney functions
- Hearing defects
- Disturbance of the hormonal metabolism
- Dermatological effects
- Suffocation and lung embolism

8**.** ***Chlorine:*** breathing small amounts of chlorine for short periods of time adversely affects the human respiratory system. Effects differ from coughing and chest pain, to water retention in the lungs. Chlorine irritates the skin, the eyes, and the respiratory system.

http://www.lenntech.com/periodic/elements/o.htm #ixzz3BjEmD8ae).

9. ***Potassium*** can affect you when breathed in. Inhalation of dust or mists can irritate the eyes, nose, throat, lungs with sneezing, coughing and sore throat. Higher exposures may cause buildup of fluid in the lungs, this can cause death. Skin and eye contact can cause severe burns leading to permanent damge. One of the quality indicators of the tobacco leaves is the level of the potassium content which effecting burning quality and decreases the quantity of the chemical oxidant used in cigarette blends (NECDET ÇAMAS *et al*., 2008).

10. ***Calcium*** is the most abundant metal in the human body: It is the main constituent of bones and teeth and it has keys metabolic functions Calcium is the most abundant metal in the human body: It is the main constituent of bones and teeth and it has keys metabolic functions.

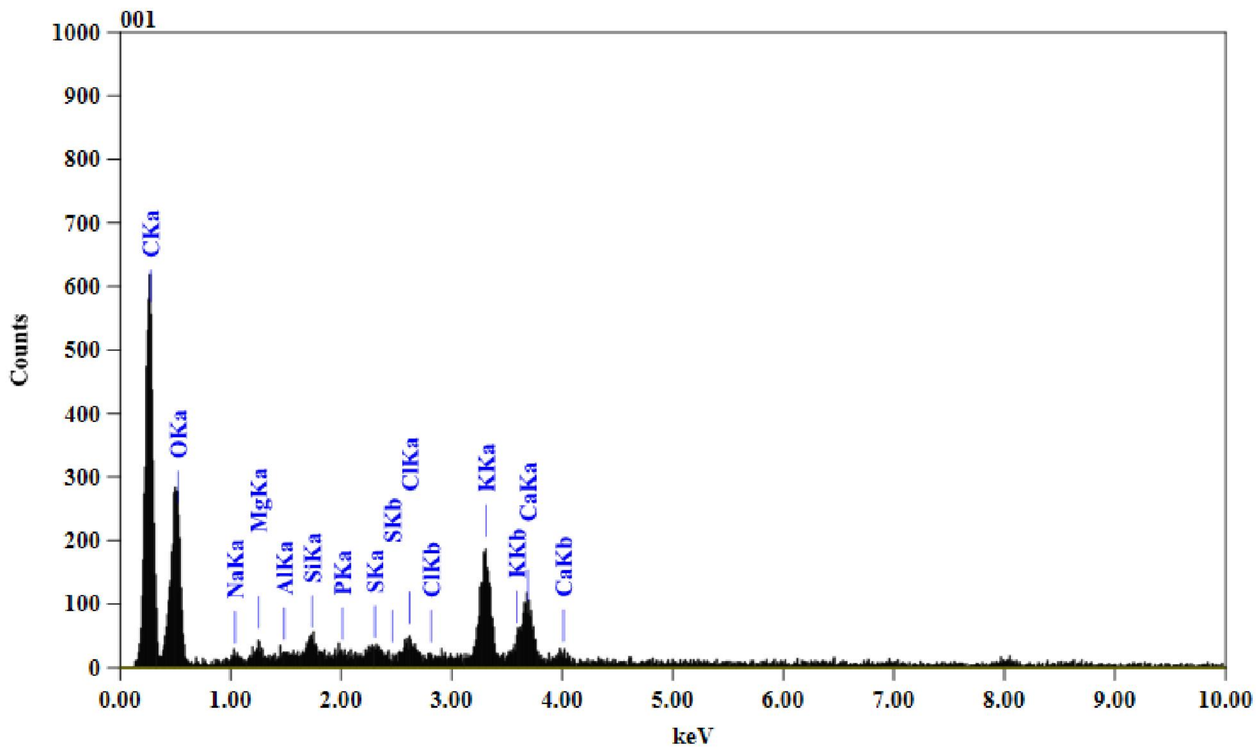
(http://www.lenntech.com/periodic/elements/o.htm#ixzz3BjEmD8ae).

For quantitative elemental analysis using SEM-EDS had revealed that the percentage of Carbon, Oxygen, Magnesium, Aluminum, Silicon, Phosphorus, Sulfur, Chlorine, Potassium and Calcium on the basis of mass % in samples as shown in table (1).

Table(1) shows that the major elements in the samples are Carbon and Oxygen, followed by Potassium, Calcium, Chlorine, Magnesium, Silicon, Aluminum, Sulfur and Phosphorus. Aluminum appears in Samples (1, 2, 4, 9, 12, 13, 14, 17) and disappears in other samples. Samples (S10),(S17) present a significant amount of Carbon and low amount of Oxygen as shown in the spectrum of Figure(3). Indicative for BC is the O/C ratio. It is 0.1 for sample S10 and S17. It is ranged from 0.65(S8) to 0.81 for sample S11. Magnesium ranged from 0.44 (S12) to 1.28(S10). Aluminum ranged from 0.0.6 (S9) to 0.11 (S12). Silicon ranged from 0.10 (S3) to 0.90 (S10, S14). Phosphor ranged from 0.09 (S1) to 0.28 (S14). Sulfur ranged from 0.11 (S1) to 0.65 (S14). Chlorine ranged from 0.37(S16) to 5.25 (S8) and calcium ranged from 1.19 (S12) to 6.07 (S10). It is clear that sample S10 high in Si, K, Ca with respect to other brands of cigarettes. Also, sample S14 high in Si, P, S with respect to other brands of cigarettes.

Table 1. The basis of mass % in samples results using SEM-EDS.

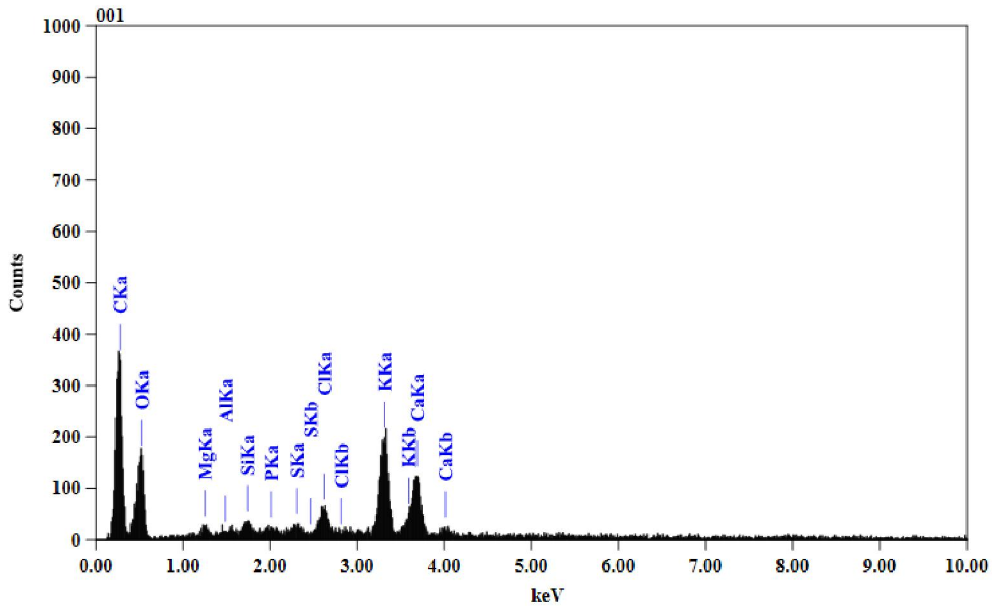
Atomic O/C ratio	Mass % Ca	Mass % K	Mass % Cl	Mass % S	Mass % P	Mass % Si	Mass % Al	Mass % Mg	Mass % O	Mass % C	Item ID
0.75	1.99	3.62	1.04	0.11	0.09	0.37	0.11	0.71	46.06	45.90	**S1**
0.79	2.82	2.80	1.13	0.34	0.10	0.17	0.16	1.13	46.76	44.58	**S2**
0.82	1.82	1.66	0.45	0.32	0.23	0.10	-----	1.10	49.23	45.08	**S3**
0.69	2.30	4.98	0.77	0.44	0.17	0.71	0.51	0.52	43.05	46.55	**S4**
0.79	1.67	4.18	0.38	0.31	0.27	0.32	-----	0.92	47.23	44.74	**S5**
0.71	2.93	3.73	1.24	0.26	0.15	0.42	----	0.83	43.95	46.48	**S6**
0.69	3.71	4.46	0.93	0.47	0.24	0.45	-----	0.94	42.48	46.31	**S7**
0.65	4.38	2.66	5.25	0.18	0.13	0.14	-----	1.26	39.92	46.10	**S8**
0.75	1.91	0.83	0.39	0.12	0.17	0.09	0.06	0.64	47.95	47.84	**S9**
0.1	6.07	8.87	1.75	0.50	0.21	0.90	----	1.28	7.13	73.29	**S10**
0.81	2.33	2.40	0.41	0.35	0.14	0.18	-----	0.65	48.43	45.11	**S11**
0.71	1.19	2.63	0.45	0.22	0.27	0.26	0.13	0.44	45.89	48.53	**S12**
0.70	1.85	2.84	0.54	0.29	0.10	0.66	0.34	0.37	45.00	48.01	**S13**
0.70	2.04	4.00	1.70	0.65	0.28	0.90	0.25	0.54	43.10	46.55	**S14**
0.66	1.96	3.49	0.77	0.17	0.13	0.45	-----	0.89	43.06	49.09	**S15**
0.75	1.42	3.04	0.37	0.25	0.16	0.42	-----	0.63	46.69	47.03	**S16**
0.1	3.73	5.77	0.72	0.37	0.21	0.89	0.28	1.02	5.77	80.24	**S17**



S17

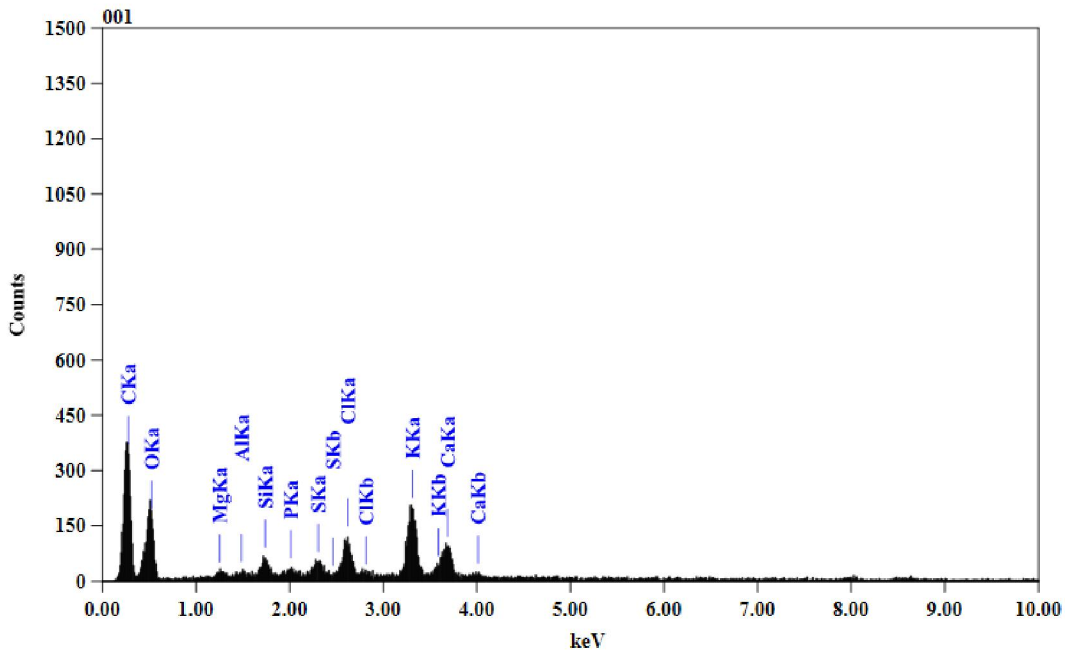
Figure (3): The x-ray spectrum for the sample S17

Below is X-ray spectra that were generated for the sample S10, S14. The Y-axis shows the counts (number of X-rays received and processed by the detector) and the X-axis shows the energy level of those counts in keV.



S10

Figure (4) : gives the x-ray spectrum for the sample S10



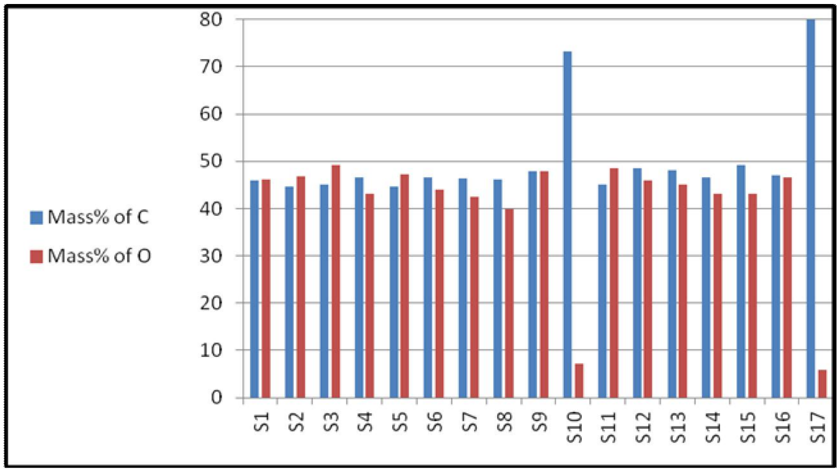
S14

Figure (5): gives the x-ray spectrum for the sample S14

Fig.6. illustrates the contents of each element in mass %. The higher values of the mass % of (C) are noted in samples S17 (80.24%) and S10 (73.29%) respectively, while the lower value of (O) is noted in sample S17 (5.77%) and S10 (7.13%). Chronic inhalation exposure to carbon black may result in damage to lungs and heart (www.disstontools.com/pdfs/DIS_MSDS1_Carbon), and if one is exposed to large amounts of oxygen for a long time, lung damage can occur. So, the excess of large amount of these two elements in cigarette Tobacco is very harmful to health (http://www.lenntech.com/periodic/elements/o.htm#ixzz3BjEmD8ae).

Figure (7) shows the mass % of K, Ca, Cl & Mg elements in all samples, while Figure (8) shows the mass % of Si, Al, S & P elements in all samples.

Figure (9). Summaries the mass % of all elements present in each sample.



Figure(6): Mass % of C & O elements in all samples

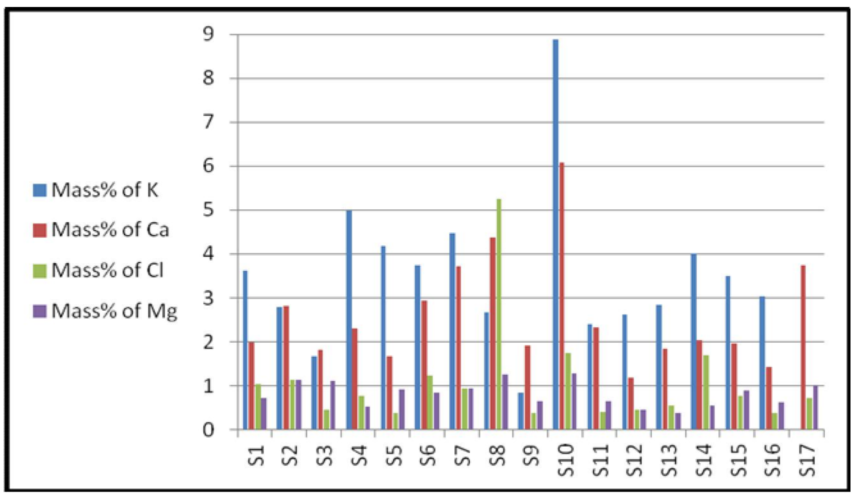


Figure (7): Mass % of K, Ca, Cl & Mg elements in all samples

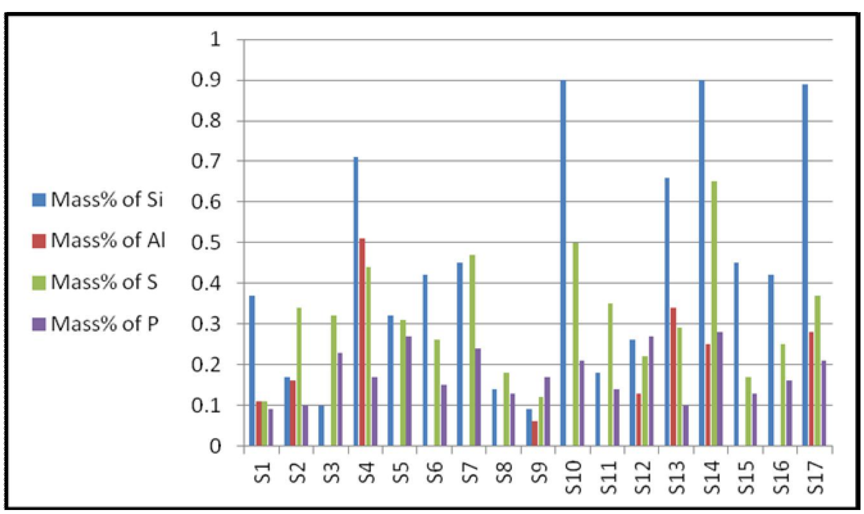


Figure (8): Mass % of Si, Al, S & P elements in all samples

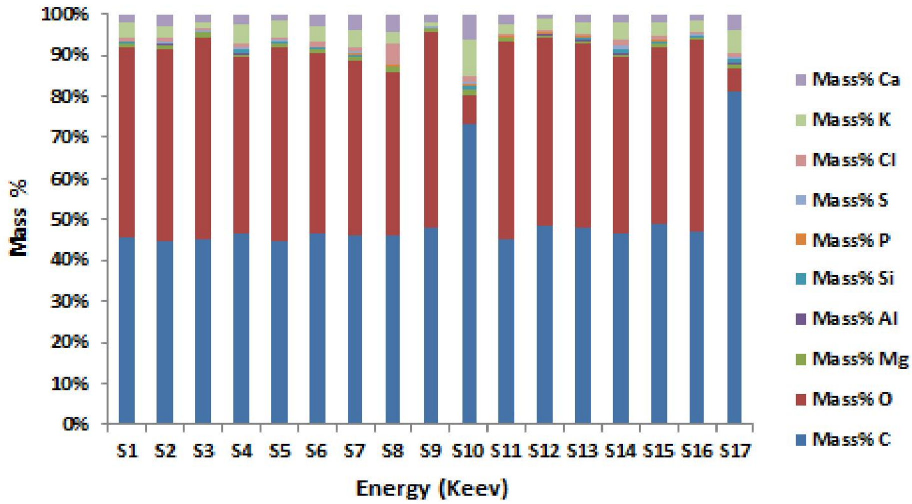


Figure (9). Mass % of all elements present in each sample

Conclusion:

Cigarette smoking is a health risk factor for many diseases. Cigarette tobacco exerts its deleterious effects not only on smokers but mostly on non-smokers and environments pollutions. Smoking is a bad habit and waste money and health. Our Islamic religion prohibits whatever destroys health. It is time to quit smoking and have a healthy community.

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