A scientific approach for evaluating extremely caked paper manuscript kept in Al-Azhar Library in Cairo

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Abstract

Purpose – Paper aims to determinate caking paper manuscript cause through studying of the manuscript components, bio-deterioration and physio-chemical deterioration factor. It will facilitate manuscripts and paper conservators to understand paper blocking and caking phenomenon.

Design/methodology/approach – The manuscript condition has been diagnosed by focusing on adhesion and fossilization regions. To achieve this, some methods of analysis and examination were used, such as visual examination, digital microscopy and scanning electron microscope were used to studying surface changes. X-ray diffraction and Fourier transform infrared microscopy were used to determinate of cellulose crystallinity, ink composition and identify the binding medium.

Findings – The results revealed the use of cotton pulp, and calcium carbonate was among the fillers that were used to improve the properties of paper. The crystallization of cellulose was lower in the first and last papers than the papers located in the heart of the manuscript. The most important reasons that led to the papers caking was the presence of fungi A. niger, Cladosporium sp, Chaetomium sp, by secreting some enzymes in combination with some other factors such as difference variation in temperature and moisture.

Originality/value – All deterioration factors participate with each other until rule the damage circle of the papers because one factor alone cannot stick the papers. It was inferred from the examinations and analyzes that were conducted for the samples.

Keywords Paper, EDX, XRD, FTIR, Caked, Fungi

Paper type Research paper

1. Introduction

The phenomenon of caked paper manuscripts is one of the most dangerous phenomena facing those responsible for the restoration and conservation of archives. Because the manuscript, in this case, has reached a state of weakness and decay that makes it difficult to take it by hand. The petrification phenomenon cannot be attributed to only one of the factors of deterioration, but rather that most of them are as if they had concluded an agreement between each other to the provisions of the circle of damage to the manuscript papers and the result of them is not only the adhesion of the papers but also that some of them it has reached the stage of petrifaction (Faubel *et al.*, 2007; Abdel-Maksoud, 2011).

The cake papers are an inescapable process (Strlic and Kolar, 2005; Jablonsky *et al.*, 2012) generated by different intrinsic factors such as sizing, filling, adhesives, the presence of acid groups, metal ions, lignin and degradation products (Havlínová *et al.*, 2009; Kolar, 1997). Besides the influence of paper composition, the lifetime of manuscripts counts largely on the quality of the storage conditions in the libraries where they are stored. Among a lot of agent's degradation, temperature, light, humidity, oxygen, pollution and microorganisms are the main environmental factors that will affect the funds of manuscript collections (Strlic and Kolar, 2005; Menart *et al.*, 2011; Qi *et al.*, 2019; Yun *et al.*, 2021). The long-term stability of paper-based artworks are determined by the extent of hydrolytic and oxidative reactions occurring during cellulose aging and which progressively reduce the physio-mechanical and chemical properties of paper, resulting finally in a total adhering of manuscript paper (Hajji *et al.*, 2016).

On the other hand, the spores of microorganisms do not grow unless they have the appropriate temperature and humidity (Hassan, 2015; Lone *et al.*, 2021). Enzymes cannot stick to papers unless some types of insects are available. For example, some insects can weave an entire manuscript in conjunction with some fungi, especially insects that dig for tunnels and that carry the fungus on their body, while the insect feeds on the leaves and digs their spending fungi secrete the enzyme that sticks to the leaves. With continued feeding and secretion, the papers turn into a solid mass, like a rock. The role of the reinforcing material in the adhesion of the papers cannot

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be hidden. It has been found that the papers found in the city of Nova Zembla, located in Russia, were covered with a layer of animal glue, and the damage to this layer resulted in the adhesion of the papers (Kathpalia, 1973).

The evolvement of specific analytical techniques enhances the procedures to document patrimonial objects made from collagen and cellulose-based materials, as well as the methods to study the influence of the environmental factors. Through the past few decades, many techniques of the analysis of paper and leather have been used for the identification of the material compounds and the assessment of the deterioration processes. For the paper, used chemical analysis for the identification of hemicelluloses, lignin and ash content in the paper (Strlič *et al.*, 2007) mentioned that the measurement of the pH of historical paper plays an important role to explain the mechanism of deterioration. Many authors used different analytical techniques for the determination of pigment used on paper manuscripts (Strlic and Kolar, 2005; Burgio *et al.*, 2008).

This study aims to identify the materials used in the manuscript analyses for the determination of paper and explain the mechanism of paper cake.

2. Historical background

This manuscript is considered one of the rare manuscripts that was given to the Al-Azhar Library by those responsible for the library of Sheikh Hassanein Muhammad Makhlouf in the village of Bani Adi in the center of Manfalut, Assiut Governorate, who occupied the position of Mufti of the Egyptian Diar in the period from 1946 to 1950 and the period from 1952 AD to 1954 AD and is one of the manuscripts that dealt with some of the topics of Islamic mysticism. The type of ink that can be traced back to the eighteenth-century AD.

3. Material and methods

3.1 Visual assessment and photography

The examination can be used with the naked eye with the help of some other means, such as magnifying lenses, to find out the reasons that led to the cake of the papers. This method is very effective because the causes and mechanisms of deterioration may be easily identifiable (Proietti and Capitani, 2004).

3.2 Digital microscopy

It is considered one of the best methods used to examine the surface appearance of paper manuscripts, so it is possible to evaluate and study the surface of paper, inks and leather cover (Tibúrcioa *et al.*, 2020). The ASIN: HJH001 B083TGGVPB digital microscope was used with a magnification range of 0x–1,600x and a lens of type 0.3 m CMOS sensor with an aperture of up to 2.0 MPIX.

3.3 Measurement of the pH

Cold extraction measurements were carried out according to Tappi method using Thermo Scientific Orion Star A111pH Benchtop Meter. The measurement was performed at the division of Pharmaceutical and Drug Industries, National Research Center, Cairo, Egypt.

3.4 Isolation and identification of fungi

3.4.1 Isolation sampling

Sterile swabs were used to wipe the surface of the paper to isolate the fungi, especially in the contaminated area. Isolation was made directly in the laboratory after the wiping process. The smears were cultivated on a general environment suitable for the growth of microbes to know the types and amount of microbial load on the archaeological samples Czapex-Dox Agar (CDA) medium, which consist of Sucrose 30.0, $NaNo_3$ 2.0g; K_2HPO_4 1.0 g; $MgSO_4.7H_2O$ 0.5 g; KCl 0.5 g, $FeSO_4.7H_2O$ 0.01 g; Agar15.0g; Distilled water 1,000 ml; pH 5.5–6. Inoculated Petri dishes with fungi were incubated at 25°C ± 2 for seven days (Gilman, 1957).

3.4.2 Purification

Fertile molds were purified by spreading a few spores upon the surface of CDA plates and incubated at 30°C for seven days. A single colony was aseptically subculture on a slant of CDA.

3.4.3 Fungal identification

Fungal isolates were identified according to morphological, cultural characteristics and microscopic examination of the fruiting bodies and spores using standard manuals for Aspergillus spp. (Diba *et al.*, 2007), Penicillium spp. (Pitt and Hocking, 1997), Cladosporium spp. (Tasic and Tasic, 2007) and Chaetomium spp. (Song and Soytong, 2017). The isolates were tested for purity and stored on slant agar at 4°C for further research.

3.4.4 Plate screening of fungal isolates for cellulolytic activity

Primary screening of fungal isolates was conducted by plating pure culture onto Czapex agar plates containing 1% (Wt./v) cellulose instead of sucrose as the sole carbon source. Mycelium discs of 5 mm size from seven days old culture was cut and one such disc was placed at the center of each agar plate. Three replicates were used for each treatment. The inoculated plates were incubated at 30°C, and the radial growth and its density were measured according to Sidkey *et al.* (1997). The most potent organisms were selected for further studies.

3.5 Investigation of the surface morphology by scanning electron microscope and energy dispersive X-ray analysis

Using SEM Model Quanta 250 Field Emission Gun (FEG) attached with Energy Dispersive X-ray Analyses (EDX Unit), with accelerating voltage 30 K.V., magnification14x up to 1000000 and resolution for Gun.1n). Samples were photographed by SEM at the Scanning Electron Microscope Laboratory, the Egyptian mineral resources authority central laboratories sector.

3.6. X-ray diffraction and energy dispersive X-ray analysis analysis of ink and pigments used

The samples of the black and red link were analyzed by X-ray diffraction (XRD) using X-ray powder diffraction measurements were performed on a D8 Discover, Bruker, a Cu Kα radiation was used as the source (λ = 1.540 Å), the measurement was performed at the Laboratory of XRD analysis Egypt Nanotechnology Center. EDX Unit, with accelerating voltage 30 K.V., magnification14x up to 1000000

and resolution for Gun.1n), the Egyptian mineral resources authority central laboratories sector.

3.7 Identification of pigment binder by Fourier's transform infrared

Using a BRUCKER VERTEX 80/80 V® spectrometer coupled with a Hyperion® microscope, Fourier's transform infrared (FTIR) transmission Spectra was introduced. All samples were scanned using Platinum diamond Attenuated Total Reflectance (ATR), with a resolution of 4 cm^{-1} in the waven number area between 4,000 and 400 cm^{-1}. FTIR was performed at the Laboratory of FTIR at the National Research Center, Cairo, Egypt.

3.8. X-ray diffraction analysis for determination the paper crystallinity

X-ray powder diffraction measurements were performed on a (D8 Discover, Bruker), a Cu Kα radiation was used as the source (λ = 1.540 Å), Egypt Nanotechnology Center, was used for determination the paper crystallinity. This method is based on analyzing the sample using XRD diffraction starting from the angle (10°–40°), then measuring the diffraction at the angle 18° and the reflection intensity at the angle of 22.6° and making a comparison between them, by using Segal crystallization (Segal *et al.*, 1962):

$$cr = 100 \times \frac{I002 - Iam}{I002}$$

where *Cr* indicates the degree of crystallization of cellulose and *I002* indicates the maximum reflection length between the angle (22°–24°) while the symbol *Iam* indicates the height of the diffraction zone (the low area between the reflection at the angle of 22°–24° and the reflection at the angle 14° –16° from the diffraction line at the 18-angle characteristic of the amorphous regions (Samsudin *et al.*, 2020).

3.9 The size of the crystal calculates

The crystal size of the cellulose was calculated using the Scherrer equation, which is an equation used to calculate the size of the nanoparticles when knowing the angle of incidence and the width of the top of one of the peaks of the diffraction pattern, and they are represented by the following relationship:

$$D002 = \frac{K\lambda}{\beta 002 \cos\theta}$$

where D represents the size of the nanoparticle, θ is the diffraction angle, K = 0.94 (correction factor), λ = 0.154 nm and b is the corrected angular width in radians at half maximum intensity of the (200) peak (Samsudin *et al.*, 2020).

4. Results and discussion

4.1 Visual assessment and photography

It was found that the papers are caked and fossilized, the cover is lost and some of the first and last pages are eroded, with some cuts and tears which may have been caused by insect damage, in addition to some spots caused by fungi, dust and dirt (Hassan, 2020) with an efflorescence of salts on some papers and loss of inks on some pages (Figures 1a, 1b and 1c).

Figure 1 Deterioration aspects found on a manuscript

Notes: (a) The papers of the manuscript are caked with their side edges; (b) missing of leather bookbinding; (c) some spots caused by fungi, dust and dirt

4.2 Digital microscopy

The front and the backend guard-leaves were exposed to severe damage. The results of the examination showed that they were exposed to adhesion, severe erosion and weak mechanical properties. Some spots resulting from some microorganisms. The results that were conducted for a group of papers by studying the cross-section, the papers caked to each other and some of them even reach the stage of petrification (Figures 2a, 2b, 2c and 2d). As for the inks, it was found that was damaged, which was represented in the presence of a loss of ink grains, big and small cracks and peeling (Figures 2e and 2f).

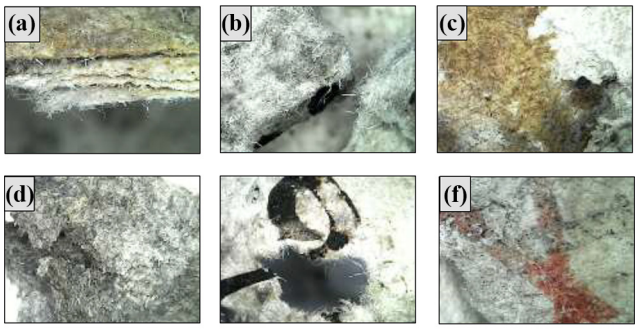
4.3 Measurement of the pH

The pH of the paper studied was 7.08. It is close to the point of neutralization, which confirms that the manuscript is made of cotton pulp, unlike papers made of wood pulp, which are characterized by an increase in the proportion of lignin, which encourages a high acidity (Koohkesh *et al.*, 2020).

4.4 Isolation and identification of fungi

The results of this study showed that the most dominant fungi on paper manuscripts were: *A. niger*, *Cladosporium sp*, *Chaetomium sp* and *Penicillium sp*. Fungi play an important role

Figure 2 Investigation of deteriorated of paper by digital microscope



Notes: (a) Cross section of a caked paper; (b) tear and cuts of paper fibers; (c) spotted fiber; (d) cracks and weaknesses of the mechanical properties of the paper fibers; (e) erosion and lose in black ink; (f) spot and lost in red ink

in the stick of missing of leather bookbinding, (C) some spots caused by fungi, dust and dirt.

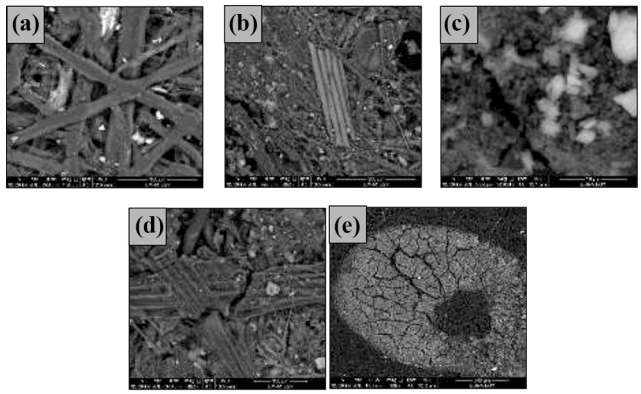
Papers of manuscripts and books (Zotti *et al.*, 2008) through the enzymes secreted by these fungi or what is known as specialized enzymes specific enzymes that can break down cellulose by cellulase enzyme and these enzymes are only very complex organic materials for the change in temperature, pH and alkalinity (Held *et al.*, 2005), the risk of fungi is that they stick to the papers of manuscripts and books in that the germs resulting from the viscous secretions of the enzymes of these fungi are growing rapidly and the rate of reproduction is very high and penetrates into the tissue of the papers causing the manuscript and the book to stick to and even hold together associated with each other until they become one sticky manuscripts block like a stone.

4.5 Investigation of the surface morphology by scanning electron microscope

The examination results revealed the scanning electron microscope for a sample of cake manuscript paper, the guard-leaf and the background that are made of cotton pulp (Zidan *et al.*, 2017) (Figure 3a). Some other materials were added to the guard-leaf such as wooden sticks (Figure 3b), and put them as they are without grinding and turning them into a pulp. Perhaps the manufacturer wanted that from thickness is greater than the thickness of ordinary paper until it performs the purpose for which it was made. It is the protective shield for the papers after the leather bookbinding, which is what it has already done. Despite losing the leather bookbinding, the remainder of the front and back lining papers have been kept between the folds of the manuscript papers (Strlic and Kolar, 2005; Mansour *et al.*, 2020), which resulted from the presence of decomposition of cellulose resulted from the breakage of the glyoxy bonds of the cellulose fibers (Zidan *et al.*, 2017), in addition to the presence of some soil and dirt (Abdel-Maksoud, 2011) (Figures 3c and d).

The black ink was exposed to damage. It was represented in large and accurate cracks, some areas covered by the ink have been caused by peeling resulting from the damage of the intermediate bonding of the ink granules represented by the Arabic gum (Remazeilles *et al.*, 2005) (Figure 3e).

Figure 3 Investigation of deteriorated of paper by SEM



Notes: (a) Original fibers paper manuscript; (b) the back guard-leaf (wood sticks and some other filling materials); (c) original paper with the growth of some microorganisms; (d) original paper with some cracks and gaps; (e) black ink with some small and large cracks

4.6. X-ray diffraction and energy dispersive X-ray analysis analysis of papers and ink used

4.6.1 Black ink

It is clear from the the analysis results that The black ink consists of $FeSO_4.6H_{2O}$ (hydrophobic iron sulfate), it has been proven that the oldest types of iron ink were prepared using iron sulfate and are known in the post-medieval period in the name of Copperas with natural tannic and Gallic acid taken from Oak–Galls (Kolar *et al.*, 2006). Ink oxidation in some places with a light color, others with a dark color and the reason for oxidative processes is due to exposure the ink has a constant difference in temperature and relative humidity, as some organic materials migrated to paper fibers (Bicchieri *et al.*, 2013; Abdel-Maksoud *et al.*, 2021).

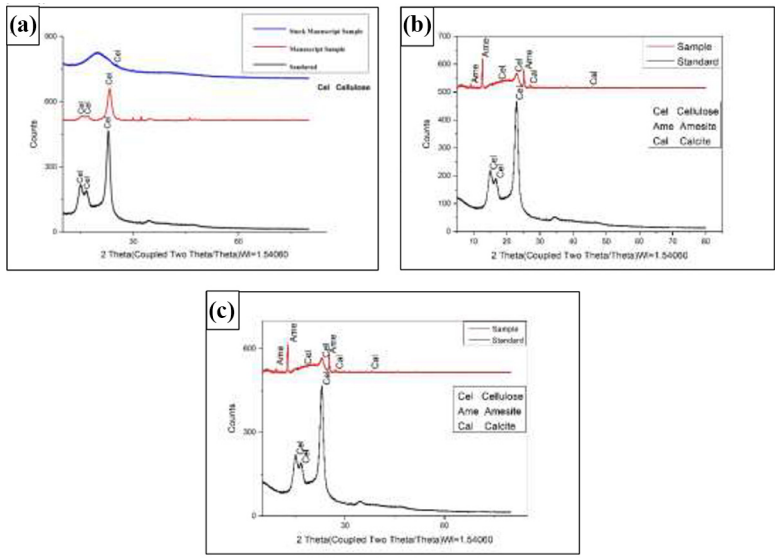
4.7. X-ray diffraction analysis for determination the paper crystallinity

4.7.1 Caked paper manuscript

It was found through the results of the analysis of the reference control of paper, original paper manuscript and caked original paper manuscript, through compensation in the Segal equation to calculate the degree of cellulose crystallization, it was found that the reference control equal to 87.64%, and by comparison with the degree of cellulose crystallization in the original manuscript paper, it was found to be equal to 87.65% and another sample of caked manuscript paper has been found to be equal to 65% (Figure 4a).

The decrease and increase in the degree of cellulose crystallization with archaeological sample can be interpretation by the effect of various deterioration factors, the rise in temperature and the decrease in moisture continuously. It leads to the destruction and damage of the amorphous areas of cellulose, which may result in an increase in the crystallized areas of cellulose at the expense of the amorphous regions, and with the presence of microorganisms, this helps the papers to cake and fossil (Samsudin *et al.*, 2020; Boukir *et al.*, 2019).

Figure 4 XRD patterns of paper crystallinity



Notes: (a) Caked paper manuscript; (b) the front guard-leaf Caked; (c) the backend guard-leaf

4.7.2 Caked guard-leaf

Through compensation in the Segal equation to calculate the degree of cellulose crystallization, it shows equal to 35.71% (Figure 4b) and the backend guard-leaf (15.62%) (Figure 4c). The decrease in the degree of crystallization of the front and back guard-leaf of caked original manuscript paper can be explained that represent the first rust wall of the manuscript from the front and backside against various deterioration factors, especially after the manuscript has lost its leather bookbinding and its papers have no cover protecting it, even to a small extent from all the different factors of damage and the deterioration phenomena's, especially after leaving for a long period of time any interference with any kind of preventive or curative conservation.

The presence of the mineral calcite in the sample is evidence that the filling material for the paper is $CaCO_3$ (Zghari *et al.*, 2018) and the presence of the mineral Amesite Mg_2 Al (Si Al) $O_5(OH)_4$ confirms that the sample has been damaged because of exposure to dust and dirt.

4.8 The size of the crystal calculates

It was found through the results of the analysis that the crystal size of the standard paper sample is equal to 7.61 nm and for the archaeological paper (6.34 nm), caked archaeological paper (1.46 nm) and caked the guard-leaf equal to 1.07 nm. It is concluded that the decrease in the crystal size of the archaeological paper and the deterioration of archaeological paper compared to the standard paper is due to the oxidation processes occurring for cellulose which led to a complete or almost complete recrystallization of the components included in the composition of cellulose (Popescu *et al.*, 2011; Hajji *et al.*, 2015).

4.9 Attenuated total reflectance-Fourier's transform infrared spectroscopy

According to Popescu (2011), Lionetto (2012), Zghari (2018) and Samsudin *et al.* (2020), the characteristic spectra of cellulose are found in the OH and CH stretching vibrations in the 3,800–2,700 cm^{-1} region and the "fingerprint" region which is assigned to different stretching vibrations of different groups from cellulose components in 1,900–800 cm^{-1} (Figures 5a, 5b and 5c).

The structural changes of cellulose feature due to the different deterioration factors that lead to adhered manuscripts papers. High temperature, moisture and bio-deterioration for an extended duration caused profound changes in the OH bonds network in the cellulose, considering that the intensity of the band related to OH stretching (3,100–3,600 cm^{-1}). The presence of intra-molecular 3,332.63 cm^{-1} and inter-molecular 3,278.36 cm^{-1} hydrogen bonding in cellulose. This resulted in a decrease in an intensity compared to the standard sample (Zghari *et al.*, 2018; Lionetto *et al.*, 2012).

The stretching bands ν (CH_2) and ν (CH) located around 2,903–2,915 cm^{-1} were slightly modified. They become less intense compared to the reference sample (Hajji *et al.*, 2016).

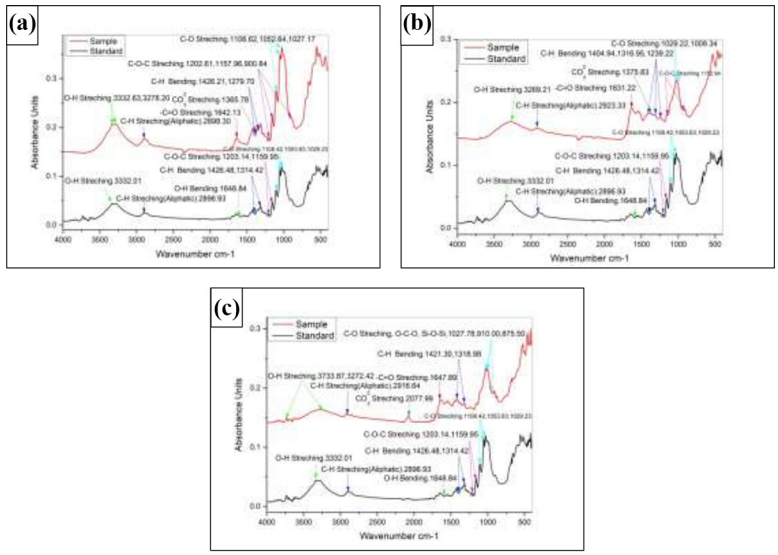
On the other hand, changes were noticed in the 400–1,800 cm^{-1} region, such as the slight decrease in the intensity of the band at 1,641–1,647 cm^{-1} characteristic of water deformation vibrations δ (OH) (Ayuni *et al.*, 2013; Hamed and Hassan, 2019).

As regard to the 900–1,200 cm^{-1} region related to the fingerprint of cellulose, it should be noted that significant changes were detected (Hajji *et al.*, 2015), specially 897 cm^{-1} region promoted the breakage of the C-O-C bonds in the β-(1, 4)-glycosidic linkage of crystalline cellulose (Zghari *et al.*, 2018; Lionetto *et al.*, 2012; Popescu *et al.*, 2011) and in pyran sugar (1,105–1,000 cm^{-1}) considered as the most sensitive to moisture and thermal perturbation (Zghari *et al.*, 2018).

4.9.1 Fourier's transform infrared spectroscopy crystalline cellulose

According to Acharya (2017), the crystalline cellulose form (strong band) absorbs at 1,425 cm^{-1} and corresponds to CH_2 symmetric bending in crystallized cellulose I. the crystallinity of

Figure 5 FTIR spectra of caked papers



Notes: (a) Caked paper manuscript; (b) the front guard-leaf Caked; (c) the backend guard-leaf Caked

caked manuscript paper index has been reported with a ratio of the peaks at I1426/I900. While the amorphous cellulose form (weak peak) shift to 1,421 cm^{-1} and 1,404 cm^{-1} in cellulose II (Ayuni *et al.*, 2013; Derrick *et al.*, 1999) for the front and backend guard-leaf. The C-H in plane bending signal at 1,375 cm^{-1} is the most appropriate for indicating cellulose crystallinity in ratio with the peak at 2,900 cm^{-1} (I1375/I2900). Referring to the recent literature report, Hajji *et al.* (2016) have assigned the band absorption at 1,317 cm^{-1} to C-H bending vibration in crystallized cellulose form, while the C-H bending vibration of amorphous cellulose form raised as a shoulder at 1,330 cm^{-1}. The cellulose contents (crystalline and amorphous) are less sensitive to the effect of changes in temperature and moisture with predominance of the crystalline phase than the amorphous one.

4.9.2 Inorganic filler

The inorganic fillers of $CaCO_3$ were investigated and identified by the broad stretching asymmetric absorption peak at 1,425 cm^{-1} and 1,375 cm^{-1} (ν_{as} C-O in CO_3^{-2}) and two weak peaks at 874 (stretching symmetric ν_s C-O of $CaCO_3$) and 710 cm^{-1} (in plane deformation δ (O-C-O) in CO_3^{-2} groups of $CaCO_3$) [45, 48] (Zghari *et al.*, 2018; Hajji *et al.*, 2015). The presence of IR inorganic filler results was also confirmed in recent works by Hajji *et al.* (2016) and Zghari *et al.* (2018) with using XRD analysis. So, in our case study, we confirmed the presence of $CaCO_3$, the high frequencies absorption bands overlapping with the cellulose bands (C-O) as reported by Hajji *et al.* (2015).

5. Conclusion

The results of this study proved the multiplicity of causes of deterioration leading to the papers caking and that one of these factors cannot cake papers alone without combining with the rest of the other factors.

The results of the microbiological examination and isolation proved that the papers were exposed to fungal infection, the most important of which was the fungus *A. niger*, which played an important role in the caking of papers.

The results of the study XRD, FTIR and EDX confirmed the examination previous results. The results showed an increase in amorphous regions from crystallized regions after comparing them with the results of standard samples.

It can be said that the caking manuscripts papers are of a special nature, and before treating and conservation them, they must be diagnosed and the reasons for their caking should be known.

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