People's Democratic Republic Of Algeria

وزارة التـــــعـــليـــــم العــــــــــــــــالي والبـــــــــحث العـــــــــــلـــــمــــــــي

Ministry of Higher Education and Scientific Research

جـــامعة سعيدة – د. الطاهر مولاي–

University Of Saïda- Dr Moulay Tahar

Technology Faculty

Department of Process Engineering

END-OF-CYCLE PROJECT

Submitted for the **Master's** Degree in **Process Engineering**

Major: Material Process Engineering

Production of a Local Polishing Material

Presented by:

Bachetoula Yasmine Farah **Labani Ibrahim EL Khalil**

Supervised, on 30/09/ 2024, in front of a jury composed of:

Dr B. Guezzen	President
Ms O. Belarbi	Supervisor
Pr M. Adjdir	Co. Supervisor
Dr A. Benhlima	Examiner

Academic year 2023/2024

Abstract
New abrasive materials for dental prosthesis polishing were developed from two residual sources: carbide lime, a by-product of acetylene production primarily composed of calcium hydroxide, and eggshell powder, which is rich in carbonates. Both materials predominantly consist of calcite and exhibit cohesive powder characteristics. After grinding, these new abrasive materials achieve a fine grain size distribution (<2mm for the eggshell powder and <5mm for the by-product). A fine mixture (<45µm) comprising 20-80% by weight of eggshell powder and carbide lime, significantly reduces surface roughness. Additionally, this mixture enhances the whiteness and brilliance of the dental prosthesis surface.

Key words: carbide lime, EggShell, abrasive, polishing, dental, prosthesis, calcite, hydroxide calcium, calcium carbonate

ملخص

تم تطوير مواد كاشطة لتلميع طقم اسنان جديدة من مصدرين رئيسيين: الجير الكربيدي، الناتج الفرعي لإنتاج الأسيتيلين والمكون بشكل أساسي من هيدروكسيد الكالسيوم، ومسحوق قشر البيض، الذي يحتوي على نسب عالية من الكربونات. تتكون كلا المادتين أساساً مر الكالسيت وتظهر خصائص مسحوق متماسك. بعد الطحن، تحقق هذه المواد الطحن الجديدة توزيعاً دقيقاً لحجم الحبيبات (أقل من 2 م لمسحوق قشر البيض وأقل من 5 م للمنتج الفرعي). خليط دقيق (أقل من 45 ميكرومتر) يحتوي على 20-80% (مسحوق قشر البيض - الجير الكربيدي) يقلل بشكل كبير من الخشونة السطحية. بالإضافة إلى ذلك، يعزز هذا الخليط بياض وتألق سطح طقم أسنان.

الكلمات المفتاحية: الجير الكربيدي، قشر البيض، كشط، تلميع، طقم الأسنان، بروتيز، كالسيت، هيدروكسيد الكالسيوم، كربونات الكالسيوم".

Résumé
De nouveaux matériaux abrasifs pour le polissage des prothèses dentaires ont été développés à partir de deux sources résiduelles : le carbure de chaux, un sous-produit de la production d'acétylène principalement composé d'hydroxyde de calcium, et la poudre de coquilles d'œufs, riche en carbonates. Les deux matériaux sont principalement constitués de calcite et présentent des caractéristiques de poudre cohésive. Après broyage, ces nouveaux matériaux abrasifs atteignent une distribution de granulométrie fine (<2 mm pour la poudre de coquilles d'œufs et <5 mm pour le sous-produit). Un mélange fin (<45 µm) composé de 20 - 80 % massique (poudre de coquille d'œuf – carbure de chaux) réduit significativement la rugosité de surface. De plus, ce mélange améliore la blancheur et la brillance de la surface de la prothèse dentaire.

Mots-clés : carbure de chaux, coquilles d'œufs, abrasif, polissage, dentaire, prothèse, calcite, hydroxyde de calcium, carbonate de calcium.

Dedication

Thank you, God, for always being by my side.

I dedicate this modest work:

To my dear Parents who supported and encouraged me throughout my school and university education, to my dear Mom, words are not enough to thank you, May God protect you and grant you happiness, health, and a long life.

To my dear sister LABANI HOUDA, who deserves all the credit for every success I've achieved.

To my beloved sisters BOUCHRA and AMINA I'm here because of you.

To DR. REDA SOLTANI, thank you for helping and guiding me all these years.

To my friends HOUARI, AHMED, and SARA, thank you for having my back all these years.

To JOURI NOUR and SEDJA to my cat thank you all

Finally, to my colleague YASSMIN, this work wouldn't have been done without you. I'm incredibly grateful to know you.

Ibrahim

Dedication:

"To my loving family, who have been my rock throughout this journey:

To my mother, whose unwavering support and guidance have inspired me to reach for the stars. Your selflessness and love have been a constant source of motivation, and I am forever grateful.

To my father, whose wisdom and encouragement have helped me navigate the challenges of academia. Your presence in my life has been a blessing, and I cherish the values you have instilled in me.

To my sisters, whose camaraderie and friendship have made this journey so much more enjoyable. Your laughter, advice, and late-night conversations have been a constant source of comfort and strength.

And to **my fellow classmates** and colleagues, who have shared in the struggles and triumphs of this academic journey. Your collective energy, creativity, and passion have created an environment that has inspired me to grow and learn.

And last but not least, I want to thank me, I want to thank me for believing in me, I want to thank me for never quitting, for being a good me.

This thesis is a testament to the power of love, support, and collaboration. I am honored to dedicate it to each and every one of you, and to myself, for believing in my own abilities and pushing through to the finish line.

Farah

Acknowledgements

First and foremost, we are grateful to "Allah" the Almighty for providing us with the health and motivation necessary to start and finish this modest job.

Without Mrs. O. Belarbi's assistance and direction, this work would not have been as rich or possible to complete. We really appreciate her for her excellent direction, her availability, her patience, and her rigor during the production of this job.

We also want to thank Pr. M. Adjdir for sharing his idea with us, working on it, and to achieve the best outcome.

We also want to thank as president of our jury Dr B. Guezzen for the honour he bestows upon us by chairing the jury of this dissertation. We also extend our sincere thanks to Dr. A. Benhlima for accepting to evaluate this work and for providing their constructive criticisms.

We really appreciate Mr. M. Alaam responsible for research and development at the ABRA-Saida company allowing us to use his facility to carry out the necessary studies.

We also would like to thank Mr. M. Diab Responsible in the quality control from the cement laboratory for his patience, assistance, and for generously sharing all the laboratory equipment with us.

Our sincere thanks to Dr. A. Medjadji and his team at the dental clinic, as well as his prosthetist colleagues, for their assistance in our experiments.

This work was carried out after obtaining permission from Linde Gas to collect samples at the Sidi Belabes unit. Therefore, we sincerely thank the management for their broad scientific mindset.

Our thanks to Mr. M. Braci from LTPO for his assistance and for providing all the necessary support to obtain the experimental results.

We acknowledge the Plateform of Industrial Technologies of Sidi Belabess University for its technical expertise of our raw materials. Pr. M. Ghaouti thank you for assist us in facilitating our access to the platform. Your support and collaboration have been instrumental in making this work possible.

Without expressing our sincere gratitude to everyone who has helped and supported us, directly and indirectly, we cannot wrap up this list of acknowledgements and thanks. We especially want to thank everyone in the process engineering department, especially Mrs. S. Lahreche and Z. Jellouli, the laboratory maintenance supervisors, for their availability and assistance.

LIST OF SYMBOLS

DM	**Dry matter**
LOI	Loss of Ignition
CI	Carr Index
HI	Hausner Index
FTIR	Fourier-Transform Infrared Spectroscopy
XRD	X-Ray Diffraction
α_r	Angle of Repose
α_m	Angle of Movement
C	Compacity
NTU	Nephelometric Turbidity Unit
CC	Cubic Centimeters

Table of Contents

Table of contents

Table of contents

List of Figures

List of Tables

INTRODUCTION

Introduction:

Materials used for artificial teeth are typically imported and expensive [1]. To achieve the desired anatomical accuracy and glossiness of tooth surfaces, both finishing and polishing materials—key abrasive procedures—are essential in the field of dentistry [2]. Each procedure relies on specialized dental abrasives. Recent advancements in abrasive technologies, as detailed in a comprehensive review by Tupinambá and al [3], have underscored the significant role of polishing agents in improving both the aesthetic outcomes and the durability of dental prosthetic materials.

Polishing abrasive have fine particles and are generally less hard than the abrasive used for finishing purposes. The polishing abrasive are responsible to smooth the surfaces which roughened typically by finishing abrasive [4]. There are some natural form of stone which are commonly used. The most common is pumice, composed of silica dioxide (SiO_2) and it is used for the polishing of tooth enamel, gold foil, dental amalgam, and acrylic resins [5]. In Algeria, the demand of the perfect aesthetic appearance is highly increasing. People are searching for the clean and beautiful teeth which will reflect their perfect smile. Therefore over 17,000 doctors and prosthesis professionals invest significantly in imported dental equipment monthly, the need for effective polishing agents is paramount [6]. It should be noted that Dental prostheses often suffer from rough surfaces due to the fabrication process [7]. Also older prostheses, especially, require restoration to minimize imperfections and improve appearance, underscoring the importance of polishing agents in dentistry [7, 8].

Introduced calcium carbonate as abrasive material has attracted interest [9]. Few studies were performed to evaluate its abrasive effect, without being excessively abrasive or damaging to gums or tooth Surfaces [10]. In another way, today, calcium hydroxide which could be obtained by hydration of carbonate calcium, is used to fill dental perforations [11].

Our key objective in this study is to develop a cost-effective polishing agent using locally sourced materials. We have selected two raw materials for this purpose: a by-product from a local company, rich in carbonate calcium and hydroxide calcium, and eggshell, recognized for its whitening and polishing properties. Both are considered as residual waste. The eggshell is a waste material available from many sources such as hatcheries, fast food restaurants, poultry industry, egg product factories and domestic homes. The industrial by-product is a carbide lime produced with a rate of 90tons in year. There is no outlet for this product.

This study focuses on the development of a startup aimed at producing a locally sourced polishing agent. It is structured into four interrelated chapters:

The first chapter provides a comprehensive overview of polishing, detailing its advantages, primary actions, and abrasive properties.

The second chapter outlines the experimental methods employed in our research.

The third chapter focuses on the characterization of our raw materials in comparison to those available on the market.

The final chapter presents a detailed analysis of the results obtained through the application of our developed polishing agent.

This research concludes with a broad summary that consolidates the key findings and scientific insights derived from our study.

References

[1] Roy, S., Maji, S., Paul, R., Bhattacharyya, J., & Goel, P. (2020). A comparison of cost and cost-effectiveness analysis of two-implant-retained overdentures versus other removable prosthodontic treatment options for edentulous mandible: A systematic review. *The Journal of Indian Prosthodontic Society*, *20*(2), 162-170.

[2] Devlin, H., & Nishimura, I. (Eds.). (2013). Oral and Cranial Implants: Recent Research Developments.

[3] Tupinambá, Í. V. M., Giampá, P. C. C., Rocha, I. A. R., & Lima, E. M. C. X. (2018). Effect of different polishing methods on surface roughness of provisional prosthetic materials. *The Journal of Indian Prosthodontic Society*, *18*(2), 96-101.

[4] Craig, R. G., O'Brien, W. J., & Powers, J. M. (2004). Dental materials: properties and manipulation.

[5] H. Z. Harraz, “Abrasive and Abrasion Minerals”, doi: 10.13140/RG.2.1.2903.6403.

[6] Alharbi, G., Al Nahedh, H. N., Al-Saud, L. M., Shono, N., & Maawadh, A. (2024). Effect of different finishing and polishing systems on surface properties of universal single shade resin-based composites. *BMC Oral Health*, *24*(1), 197.

[7] Powers, J. M., & Wataha, J. C. (2012). *Dental materials-e-book: Properties and manipulation*. Elsevier Health Sciences.

[8] Al-Rifaiy, M. Q. (2010). The effect of mechanical and chemical polishing techniques on the surface roughness of denture base acrylic resins. *The Saudi dental journal*, *22*(1), 13-17.

[9] Farghal, N., & Elkafrawy, H. (2020). The effects of activated charcoal and calcium carbonate based toothpastes on surface properties of composite resin restorative materials. *Egyptian Dental Journal*, *66*(4-October (Fixed Prosthodontics, Removable Prosthodontics and Dental Materials)), 2431-2438.

[10] Pertiwi, U. I., Eriwati, Y. K., & Irawan, B. (2017, August). Surface changes of enamel after brushing with charcoal toothpaste. In *Journal of Physics: Conference Series* (Vol. 884, No. 1, p. 012002). IOP Publishing.

[11] Gauthier, L. (2018). Les réactions du complexe pulpo-dentinaire suite à la mise en place de matériaux de coiffage indirect sur dent permanente : revue systématique de littérature, thèse de doctorat, Université de Marseille.

CHAPTER I

GENERALITIES ON THE PROCESS OF DENTAL PROSTHESES POLISHING

1. Introduction

The process of polishing dental prostheses involves several critical steps to achieve a smooth and glossy finish while preserving the original contours. This chapter provides an overview of the literature on abrasive materials used for polishing and their application in dental laboratories. Additionally, it introduces and reviews potential materials that could be utilized as abrasive agents.

2. Polishing Techniques:

Polishing is a technique for removing scratches by replacing them with finer ones. It involves using materials such as pumice, revolving wheels, and polishing compounds to achieve a glossy surface [1]. During the polishing process, the labial, buccal, lingual, and palatal external surfaces of the denture are polished with wet pumice on a rag wheel attached to a dental lathe operating at a slow speed [1]. The polished surface should extend just over the borders without reducing their height or width.

2.1. Principle:

To improve appearance, reduce adhesion, and prevent corrosion, dental prosthesis surfaces should be polished [2]. Polishing effectively removes plaque, biofilm, and stains [3], resulting in a glossy, lustrous finish. Various techniques and materials are employed for polishing, with patient comfort and care as top priorities. Over time, techniques have evolved to place greater emphasis on meeting patient expectations and providing effective, individualized polishing treatments. Therefore, the objectives of the polishing process can be summarized as follows: [4]

Shine: The natural gloss improves the prosthesis's appearance and adds to its overall realness.

Color: matching colors to produce a natural look.

The polishing process eliminates the need for a laborious and time-consuming mechanical polishing sequence by introducing a chemical polishing method. This method provides a smooth surface without requiring multiple steps, thereby enhancing efficiency in the finishing process [5].

2.2. Polishing action:

The experimental situations encountered in finishing and polishing techniques are two-body abrasion and three-body abrasion. These are the most widely accepted terms for the classification of the abrasive-wear modes.

2.2.1. Two-body abrasion:

The concept of two-body abrasion in polishing involves abrasive particles that are firmly fixed to a substrate, such as a dental bur, disk, wheel, or strips [6].These particles directly interact with the surface being polished, creating abrasion through direct contact. This method does not require a polishing paste (figure I-1). Therefore, in the context of polishing, two-body abrasion refers to direct contact between abrasive particles and the surface being polished.

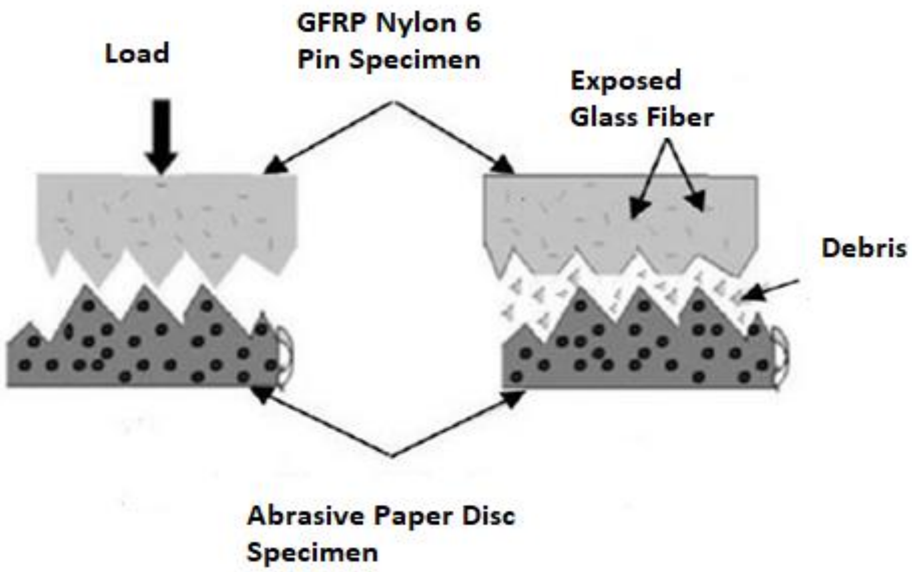


Figure I-1: Concept of two-body abrasion [7].

2.2.2. Three-body abrasion:

The concept of three-body abrasion in polishing involves abrasive particles moving between the surfaces being polished and the tool or medium applying the abrasion [8]. Unlike two-body abrasion where abrasive particles are fixed to the substrate, in three-body abrasion, the abrasive particles are free to move, enhancing the polishing action (figure I-2) [6]. An example of three-body abrasion is polishing with a rubber cup and prophylaxis paste in dentistry [8], where abrasive particles mixed in the paste move between the tooth surface and the rubber cup, creating a polishing effect .

. Dental hygienists commonly use three-body abrasion. Dental hygienists frequently employ this kind of abrasion for efficient polishing.

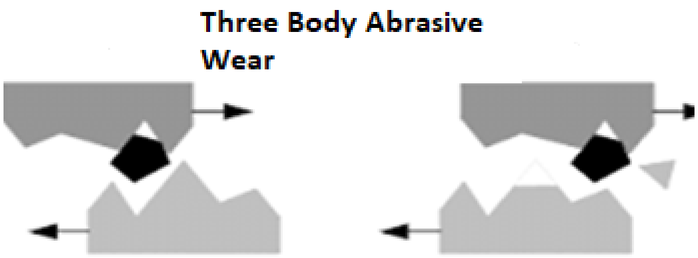


Figure I-2: Concept of three-body abrasion [8].

3. Abrasion

The abrasion process is a complex mechanism that occurs when two materials come into contact, where a material with high hardness scratches the surface of a less hard material.

Abrasion generally occurs due to friction and pressure forces exerted on the surface of the material. [9].

Irreversibility is a key characteristic of abrasion. Once the surface of the material has been changed, it is difficult, if not impossible, to completely restore its original shape and structure.

3.1. Factors influencing abrasion:

Abrasion appears to be influenced by many factors, with the most significant being the abrasive particles, particularly their size and sharpness.

3.1.1. Particle Size:

Particle size is directly proportional to the quality of the surface roughness [10, 11]. Also, the strength of an abrasive material relates to the hardness of the material. Hardness could be defined as the resistance of a material before it is plastically deformed by being indented or scratched by another material. Hence, if an abrasive material is too hard, or if the particle size is very coarse, the quality of the surface roughness will be affected as deep scratches result [12]. For example, smaller particle sized abrasive materials facilitates the polishing mechanisms by rapidly wearing away surfaces to expose newly-formed sharp materials needed to decrease surface roughness [13].

3.1.2. Particle Shape:

An important factor in the polishing process is the shape of the abrasive particles. Compared to spherical particles, angular particles are more aggressive and remove material at a faster rate. The polishing efficiency of angular particles is higher than that of spherical particles due to their sharper edges and corners, which can more easily penetrate the material surface. As the aspect ratio of the particles increases, the surface roughness decreases, suggesting that longer particles result in a finer surface finish. An abrasive material does not have the correct particle shape, or does not break down into newly sharp-edged particles, the quality of the polishing materials will be compromised [14]. Irregular sharp particles abrade surfaces more rapidly than more rounded particles, which leave duller cutting angles [15].

3.2. Abrasives materials:

Abrasives can be of natural or synthetic origin and differ mainly in their degree of hardness. The hardness of abrasives is measured using special scales such as the Knopp scale or the Mohs scale. Diamond is the hardest known natural abrasive material, but there are also other natural and synthetic materials such as silicon carbide, corundum, and zirconium [16]. To manufacture an abrasive, it is necessary to have a support to which the abrasive powder can adhere. These abrasive instruments are used in a variety of applications for sanding, polishing, and other finishing operations [17-18].

Usually, abrasives are classified into two types: natural and synthetic.

3.2.1. Natural abrasives:

In the early days of mechanical manufacturing, the abrasives used were mainly of natural origin, in particular:

- **Emery:** it is a variety of corundum. When ground, it makes it possible to obtain a powder used as an abrasive. Its hardness is 9 on the Mohs scale. Ex: emery paper, Emery canvas... [19]
- **Flint:** its hardness is 7 on the Mohs scale. [17]
- **Garnet:** from the mineral family, its hardness is 7 — 7.5 on the Mohs scale. [16]

3.2.2. Manufactured/synthetic abrasives:

With technological advancements, the introduction of the first synthetic abrasives marked a turning point in the industry. These new products offered improved performance and a wider variety of options for abrasion tasks. [19]

Synthetic abrasives are artificially manufactured materials used in a variety of abrasive processes. These synthetic abrasives are widely used in industrial applications such as grinding, polishing, pickling and sanding. In addition, synthetic abrasives can be formulated to meet specific requirements, making them suitable for a wide range of materials and surfaces to be treated [17]. They are used in powder form.

3.3. Abrasives in dental field:

Abrasives are an integral part of oral health products and play an essential role in toothpastes and professional dental polishing materials. Their main function is to facilitate the removal of plaque stains and tartar from the teeth, thus significantly to the prevention of caries and gum disease [21]. The abrasive materials are mixed with water to obtain polishing slurry, used to get rid of external stains and leaving the enamel glossy and smooth. In dental treatment, some typical polishing slurries are as follows:

Prophy paste: is a classic polishing paste available in a variety of tastes such as mint.

Feldspar: A purifying substance combined with water to form a slurry or paste for polishing.

Sodium-aluminum silicate: A polishing chemical that leaves enamel glossy and smooth after removing external stains.

However, pumice is the most abrasive substance used to polish in dental field.

3.3.1. Pumice:

Pumice is a cheap and abundant vesicular rock of volcanic origin, formed by the combination of SiO_2 and Al_2O_3 at variable proportions, with trace amounts of other oxides and some aluminum oxide between the vesicles, glassy material forms thin partitions, threads, and fibers [**22**]. The vesicles, or cavities, might have a tubular, elongated, or spherical shape based on how the solidifying lava flows through them

The natural variability of pumice's composition allows for a wide range of physical and chemical properties, which provides the opportunity for upgrading this mineral resource [23].

Pumice stone has amorphous structure. This volcanic glass is amorphous and referred to as "mineraloid." The material cools so quickly that atoms in the melt are not able to arrange themselves into a crystalline structure.

In dentistry, pumice stones are frequently used for a number of reasons, such as: [24]

Cleaning and polishing teeth: Teeth are made clean and shiny by using a pumice stone to remove stains, plaque, and tartar.

Dental restorations: Teeth are prepared for restorations including fillings and crowns using a pumice stone.

Dental hygiene: Dental equipment and instruments can be cleaned and polished with a pumice stone.

3.3.2. Calcium carbonate:

Calcium carbonate, represented by the chemical formula $CaCO_3$, is a versatile compound present in various natural reservoirs such as rocks, shells, and pearls. It serves manifold purposes, notably as a key constituent of limestone, chalk, and marble [25]. Pervious study also detected a significant increase in composite surface roughness after using calcium carbonate tooth paste [26]. Carbonates were less abrasive compared to the ones containing bicarbonate [27]. lts crystalline structure is calcite ($CaCO_3$), which has a trigonal crystal structure.

Additionally, it serves as a calcium source in animal feed, food, and pharmaceuticals [28]. Medically, calcium carbonate serves as both a calcium supplement and an antacid [28].

Calcium carbonate materials are correlated to its morphology and particle size. For example, scalenohedral-shaped calcium carbonate particles tend to have small particle sizes and provide relatively insignificant cleaning effectiveness. On the other hand, large rhombohedral-shaped (sometimes referred to as “cubic') calcium carbonate particles have increased cleaning and abrasive benefits, but often abrade all too well: their abrasiveness leading to a concern for possible damage to teeth and gums. Thus, calcium carbonate material must be sufficiently abrasive to effectively clean, but must not be so excessively abrasive that it may damage tooth and Soft tissue Surfaces. [29]

3.3.3. Calcium Hydroxide:

Calcium hydroxide, represented by the chemical formula $Ca(OH)_2$, is a mineral compound comprising calcium cations and hydroxide anions. Commonly known as slaked lime or hydrated lime, such as nitrogen and sulfur oxides. Calcium hydroxide is also utilized in dental treatments, including root canal fillings [30]. Calcium hydroxide, a versatile material introduced to dentistry in the early 20th century, serves multiple purposes [31]. Its high pH and antibacterial properties make it valuable for stimulating mineralization and treating various dental issues [32]. Different formulations target specific therapeutic actions, with outcomes influenced by the targeted tissues [31].

3.3.4. Limestone:

The main component of limestone, a sedimentary rock, is calcium carbonate ($CaCO_3$) in the form of calcite minerals. It is a typical mineral that can be found in igneous, metamorphic, and sedimentary rocks. Scalenohedral, rhombohedral, and tabular shapes are among the many forms that calcite crystals can take on [33].

Because limestone is strong and long-lasting, it is utilized as a filler in dental restorative materials including composite resins and cements. To assist remove surface stains and plaque from teeth, toothpaste contains limestone as an abrasive. To increase the biocompatibility and Osseo integration of dental implants, limestone is applied as a coating material. In orthopedic and dental applications, limestone is utilized as a bone graft material to encourage the formation and regeneration of new bone. [34]

3.3.5. Eggshells as abrasive material:

Calcium carbonate ($CaCO_3$) crystals represent the majority of the material that makes up an eggshell [35]. Similar to dental enamel, eggshells are hard but brittle. There are resistant to crushing from top to bottom because of their distinctive form [36].

There are several different crystal habits that calcite in eggshells can display, such as scalenohedral, rhombohedral, and tabular shapes. Calcite crystals in eggshells can range in size and form from 1 to 10 μm in diameter, however they can be any size [37].

Eggshell powder has been researched as a potential material for bone grafts and the regeneration of teeth, tendons, and cartilage [38]. It has been demonstrated that the calcium carbonate in eggshells helps to fortify tooth enamel and aid in the restoration of broken teeth. Because eggshells are biocompatible and can stimulate bone formation, there has been research on using them as a possible material for dental implants [39].

References:

[1] Jefferies, S. R. (1998). The art and science of abrasive finishing and polishing in restorative dentistry. *Dental Clinics of North America*, *42*(4), 613-627.

[2] Rashid, H. (2014). The effect of surface roughness on ceramics used in dentistry: A review of literature. *European journal of dentistry*, *8*(04), 571-579.

[3] Michael Z (2012), *the effect of clinical polishing protocols on ceramic surface texture and wear rate of opposing enamel: laboratory study*, thesis of doctor of clinical dentistry (prosthodontics), University of Adelaide, Australia

[4] Al-Rifaiy, M. Q. (2010). The effect of mechanical and chemical polishing techniques on the surface roughness of denture base acrylic resins. *The Saudi dental journal*, *22*(1), 13-17.

[5] S El-Din, M., Badr, A. M., Agamy, E. M., & Mohamed, G. F. (2018). Effect of two polishing techniques on surface roughness of three different denture base materials (an in vitro study). *Alexandria Dental Journal*, *43*(3), 34-40.

[6] Caren M. Barnes, R.D.H., M.S, Polishing Materials and Abrasion, published on websitehttps://pocketdentistry.com/polishing-materials-and-abrasion

[7] Kumar, S., & Panneerselvam, K. (2016). Two-body abrasive wear behavior of nylon 6 and glass fiber reinforced (GFR) nylon 6 composite. *Procedia Technology*, *25*, 1129-1136.

[8] Cheng w, Dong s, (2022), Tribological characteristics of three-body abrasive wear on MoS2 films, .School of Mechatronics and Vehicle Engineering, Chongqing Jiaotong University, China

[9] Callister, W. D., & Rethwisch, D. G. (2018). Materials Science and Engineering: An Introduction. Wiley.

[10] Rizzatti-Barbosa, C. M., Gabriotti, M. N., Joia, F. A., Machado, C., & Ribeiro, M. C. (2006). Surface roughness of acrylic resins processed by microwave energy and polished by mechanical and chemical process. *Brazilian Journal of Oral sciences*, 5(16), 977-981.

[11] Jassem, W., Al Furaiji, N., & Qasim, S. (2024, May). Evaluation of surface roughness of polymethyl methacrylate reinforcement with nano alumina after polishing with different abrasive materials. In *AIP Conference Proceedings* (Vol. 3097, No. 1). AIP Publishing.

[12] Ahmed Areeg, Shihab (2011). Evaluation and compare between the surface roughness of acrylic resin polished by pumice, white sand and black sand. *Journal of Kerbala University*, 9(1), 49-54.

[13] Onyia, J. C., Ajaegbu, E. E., Okike, B. M., Ikuesan, A. J., Bello, A. M., Okolo, I. O., Ezeh, Ukachukwu C.; Dieke, Adaobi J.; Onuora, Adaora L.; Ezugworie, Flora N.; Izekor, Ese S.; Ndubuisi, Juliana O.; Adimora, Ethel E.; Nwigwe, Juliet O.; Iloabuchi, Lucy C.; Bassey, Nnyeneime U.; Nduka, Florence O & Ifeloju, T. O. (2022). Evaluation of Gallus gallus domesticus Eggshell and Glass as an Alternative to Dental Pumice. *Tropical Journal of Natural Product Research*, 6(11).

[14] Van Noort, R., & Barbour, M. E. (2023). *Introduction to Dental Materials-E-Book: Introduction to Dental Materials*-E-Book. Elsevier Health Sciences.

[15] Hassan, U., Farooq, I., Moheet, I. A., & AlShwaimi, E. (2017). Cutting efficiency of different dental materials utilized in an air abrasion system. *International Journal of Health Sciences*, 11(4), 23.

[16] R. S. Roth A Review of Their Properties and Applications, *Journal of the American Ceramic Society*, 1964, 47(10): 531-536

[17] R. L. Folk A Review of Their Properties and Applications, *Journal of Sedimentary Petrology*, 1965, 35(2): 341-348

[18] R. A. Laudise a Review of Their Properties and Applications & quot; by, *Journal of Crystal Growth*, 1979, 46(2): 151-164

[19] W. D. Kingery a Review of Their Properties and Applications; *Journal of the American Ceramic Society*, 1960, 43(10): 513-518

[20] Nedjai a, Krim h, (2020), Study of vitrified binders applied to abrasive wheels of the ABRA-Saida unit. Master's thesis (Saida University)

[21] Abrasives in Dentistry: A Review of Their Role in Oral Health" by J. M. Powers, *Journal of Dental Research*, 2013, 92(9): 753-761

[22] Mourhly, A., Khachani, M., Hamidi, A. E., Kacimi, M., Halim, M., & Arsalane, S. (2015). The synthesis and characterization of low-cost mesoporous silica SiO2 from local pumice rock. *Nanomaterials and Nanotechnology*, 5, 35.

[23] Shiferaw, W. O. L. E. L. A. (2020). *Experimental investigation on lime stabilized volcanic pumice subbase* (Doctoral dissertation, MS Thesis, Addis Ababa Science and Technology University).

[24] Firoozeh, F., Zibaei, M., Zendedel, A., Rashidipour, H., & Kamran, A. (2013). Microbial contamination of pumice used in dental laboratories. *Healthcare in Low-resource Settings*, 1(1), e5-e5.

[25] Al Omari, M. M. H., Rashid, I. S., Qinna, N. A., Jaber, A. M., & Badwan, A. A. (2016). Calcium carbonate. *Profiles of drug substances, excipients and related methodology*, 41, 31-132.

[26] Farghal, N., & Elkafrawy, H. (2020). The effects of activated charcoal and calcium carbonate based toothpastes on surface properties of composite resin restorative materials. *Egyptian Dental Journal*, 66(4-October (Fixed Prosthodontics, Removable Prosthodontics and Dental Materials)), 2431-2438.

[27] Pertiwi, U. I., Eriwati, Y. K., & Irawan, B. (2017, August). Surface changes of enamel after brushing with charcoal toothpaste. In *Journal of Physics*: Conference Series (Vol. 884, No. 1, p. 012002). IOP Publishing.

[28] Erdogan, N., & Eken, H. A. (2017). Precipitated calcium carbonate production, synthesis and properties. *Physicochemical Problems of Mineral Processing*, 53.

[29] Liu, S.-T., Martin, M. J., Fultz, W. C., & McGill, P. D. (2006). Precipitated calcium carbonate (U.S. Patent No. 6,989,142). U.S. Patent and Trademark Office.

[30] Fellahi, S., & Sid, R. (2022). *Stepwise excavation (SW) et partial removal caries (PRC) dans les caries profondes* (Doctoral dissertation, Université Constantine 3 Salah Boubnider, Faculté de médecine).

[31] Nishanthi, R. (2021). Role of calcium hydroxide in dentistry: A review. *International Journal of Pharmaceutical Research*, 12(2), 377-382.

[32] Benoist, F. L., Ndiaye, F. G., Kane, A. W., Benoist, H. M., & Farge, P. (2012). Evaluation of mineral trioxide aggregate (MTA) versus calcium hydroxide cement (Dycal®) in the formation of a dentine bridge: a randomised controlled trial. *International dental journal*, 62(1), 33-39.

[33] Oates, J. A. (2008). *Lime and limestone: chemistry and technology, production and uses*. John Wiley & Sons.

[34] Skinner, H. C. W. (2012). Minerals and human health. In D. J. Vaughan & R. A. Wogelius (Eds.), Environmental Mineralogy II (Vol. 13, pp. 0). Mineralogical Society of Great Britain and Ireland.

[35] Mohadi, R., Anggraini, K., Riyanti, F., & Lesbani, A. (2016). Preparation calcium oxide from chicken eggshells. Sriwijaya *Journal of Environment*, 1(2), 32-35.

[36] Nasif, R. A. (2015). Preparation and characterization of eggshell powder (ESP) and study its effect on unsaturated polyester composites material. *Iraqi journal of applied physics*, 11(1).

[37] Gaber, M. A., & Gaber, M. (2020). A new approach of Egyptian calcium carbonate utilization as ingredients of toothpaste manufacture. *Egyptian Journal of Geology*, 64, 329-336.

[38] Hincke, M. T., Nys, Y., Gautron, J., Mann, K., Rodriguez-Navarro, A. B., & McKee, M. D. (2012). The eggshell: structure, composition and mineralization. *Front Biosci*, 17(1), 1266-1280.

[39] Kadhim, D. R., Hamad, T. I., & Fatalla, A. A. (2022). Use of eggshells as bone grafts around commercially pure titanium implant screws coated with nano calcium sulfate. *International Journal of Biomaterials*, 2022(1), 8722283.

CHAPTER II

EXPERIMENTAL PROTOCOLS

1. Introduction

This chapter deals with the principles and procedures of the preparation and characterisation of the materials used in this study.

We will look at methods dedicated to structural characterizations such as X Ray Diffraction. Then, we will focus on, density measurements, specific surface area measurements, and particle size measurements. We have also characterized our new abrasive materials using Fourier-transform infrared spectroscopy.

The various analyses described in this paragraph are carried out in the Process Engineering Department, LTPO Laboratory, and Control Quality Laboratory of the Cement Plant Hassassna.

2. Humidity

Humidity typically refers to the moisture content or moisture level within a material or substance. It's a measure of how much water or moisture is present within a solid material, which can be important in various industries and applications

Procedure

Watch glass is placed with 2 g of material in a drying oven for 24 hour at 105°C.

The humidity of the sample is given as below

$$\textbf{Humidity (\%)} = \textbf{Wwet} - \textbf{Wdry} / \textbf{Wwet} * \textbf{100} \quad \ldots\ldots\ldots\ldots\ldots \textbf{(1)}$$

Where

Wwet: Initial weight of the wet sample (g)

Wdry: Final weight of the dry sample (g)

3. X-ray fluorescence spectrometry

X-ray fluorescence spectrometry is a comprehensive elemental analysis technique for identifying and determining most of the chemical elements in a sample.

The sample to be analyzed is placed under a beam of X-rays in a pellet. Under the effect of these X-rays, the atoms making up the sample change from their ground state to an excited state. The excited state is unstable, and the atoms tend to return to their ground state, releasing energy in the form of X-ray photons.

The X-ray fluorescence spectrometer used is a PAN analytical ZETIUM (figure II-1)

Figure II-1: X-ray fluorescence spectrometer PAN analytical ZETIUM

4. Loss on ignition determination (LOI) [1]

The principles of loss on ignition analysis often include elements such as determining the time required for the material to reach certain critical temperatures, measuring physical or mechanical changes during exposure to heat, and overall assessment of the material's performance under fire conditions.

Procedure

1g of sample is added in a weighted crucible which is placed in a calciner for 30 minutes at 925°C (figure II-2). The crucible is finally weighted Loss on ignition (LOI) rate is given by the following relationship:

$$\mathbf{LOI\ \% = (W1-W2) * 100 \ldots\ldots\ldots.. (2)}$$

Such as:

W1: mass of crucible with 1 g of sample (g).

W2: mass of cooled crucible (g).

Figure II-2: Controlab calciner

5. X-Ray diffraction

The phenomenon known as X-ray diffraction (XRD) occurs when a crystal's uniformly spaced atoms cause an interference pattern to appear in the waves included in an X-ray incident beam. The way the crystal's atomic planes interact with the X-rays is identical to how an evenly governed diffraction grating interacts with a light beam.

Powder X-ray diffraction data was collected using a Rigaku MiniFlex 600 powder X-ray diffractometer (University of Tiaret) equipped with Cu Kα radiation (λ = 1.5406 Å, Gemonochromator). A Bragg angle 2 θ ranging from 10° to 80° with a 0.02° step was employed.

6. Infrared Spectroscopy

By measuring how much infrared light is absorbed by the molecules in a sample, a non-destructive analytical method called infrared spectroscopy (IR) can be used to determine and quantify the molecular structure of the sample. It is predicated on the idea that molecules absorb particular infrared radiation frequencies that correlate to their vibrational modes. Functional groups, molecule bonds, and molecular conformations can all be identified and examined using the distinct fingerprint of the resulting infrared spectrum.

Procedure

The sample is prepared by ensuring it is dry and free of contaminants, then placed in the IR spectrometer to record the IR spectrum. The spectrum is analyzed to identify functional groups, determine molecular structure, and quantify the concentration of specific compounds in mixtures. Finally, the results are interpreted, using the unique IR spectrum as a molecular fingerprint to identify the sample and determine its composition.

The FT-IR spectra were obtained with a Fourier transform infrared spectrometer (genesis II DTGS- University of Tiaret). The scanning wavelength of infrared was 4000–400 cm^{-1}

7. Granulometric analysis

The principle of particle size analysis is based on separating the particles of a material into distinct fractions according to their size. This is usually done using sieves with different mesh sizes (figure II-3). Sieves have defined mesh sizes, enabling particles above the respective mesh to be retained. The resulting particle size distribution gives a quantitative representation of the different particle sizes present.

Figure II-3: Sieves with different mesh sizes

Particle size analysis enables the determination of both the average grain size and the size distribution within the material. We employed two distinct methodologies for particle size determination:

• Particle size analysis by sieving (method: dry after washing) for particle size fractions greater than 80 µm.

• Particle size analysis by sedimentometry for particle size fractions less than 80 µm.

7.1. Granulometric analysis (method: dry after washing) [2]

This standard applies to the elements of a natural soil passing through the 80µm sieve. It concerns soils with a particle size of less than 80 µm. Analysis is carried out by sedimentation in water, by measuring the speed at which grains fall under the effect of gravity, according to Stokes' law. However, particles smaller than 1 µm cannot be differentiated using this test.

Procedure

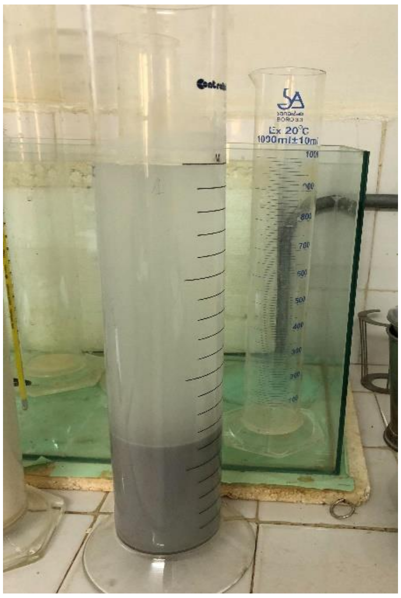
A representative sample of the material to be analysed is selected. The sample is dried at a controlled temperature to remove moisture. The coarsest sieve is placed at the bottom of the sieve stack. The sample is placed on the coarsest sieve. The sieves are assembled in descending order of mesh size. The sieves are shaked mechanically or manually for a set time to separate the particles by size. Each fraction of material collected on the collection trays are Weighted.

7.2. Granulometric analysis by sedimentation [3]

Granulometric analysis by sedimentation is used to determine the weight distribution of the size of soil particles smaller than 80 micrometres. It complements sieve analysis (standard NF P 94-056) for the finest elements in natural soil.

Procedure

A 500 g sample is submerged in water in a tank for 24 hours. The sample is then washed using a siphon and an 80 µm sieve. The sieve is subsequently dried in an oven at 105°C for four hours. After drying, the sample is sieved again to break it up and ensure homogeneity.
An 80 g test portion is then taken from the sample. This test portion is placed in a container suitable for use with a mechanical stirrer.
To this container, 500 milliliters of a 5% sodium hexametaphosphate ($Na_6P_6O_{18}$) solution is added as a dispersant. The sample is allowed to soak in the dispersant for a minimum of fifteen hours. Finally, a mechanical stirrer is used to disperse the suspension for at least three minutes at 1000 rpm. Not be disturbed, a 2-liter graduated cylinder is filled with the suspension. The cylinder is then topped off with room-temperature distilled water up to the 2-liter mark (figureII-4).



sedimentation tube filled **sedimentation tube filled after 24h**

Figure II-4: Granulometry analysise by sedimentometry

We use a second 2-liter test tube filled with the same distilled water as the first one as a control. The hydrometer and thermometer are fully submerged in water. The suspension is vigorously shaken vertically using a manual stirrer. The hydrometer is then submerged, the manual stirrer is removed, and the stopwatch is started immediately. Readings are taken at predetermined intervals.

The proportion of particles (P) with diameters smaller than 80 µm is calculated by a software.

8. Hydrogen potential (pH) measurement

pH or Hydrogen potential is a measure of the acidity or basicity of a medium. More precisely, pH represents the concentration of hydrogen ions (H^+) in a solution. It is given by the following relationship:

$$pH = - Log [H^+] \ldots\ldots\ldots\ldots\ldots (3)$$

The pH meter used is a Consort C863 (figure II-5)

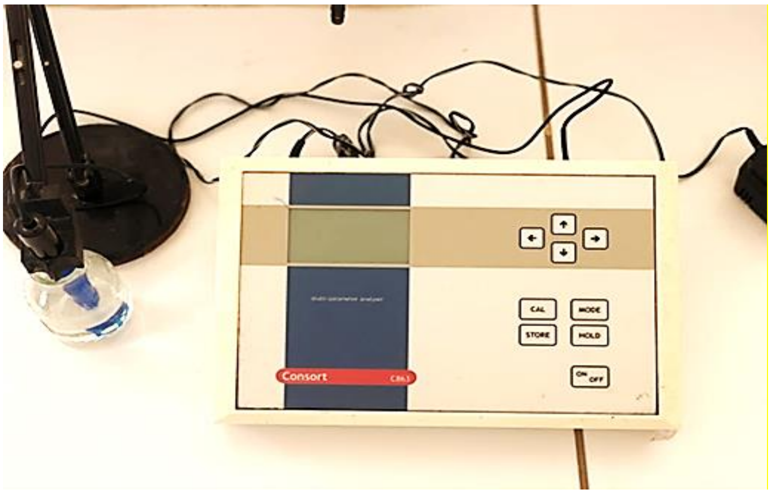


Figure II-5: Hydrogen potential Consort C863

9. Conductivity measurement [4]

Electrical conductivity is a measure of a substance's ability to conduct an electric current. The conductivity meter used is HANNA conductivity meter EC 215 (figure II-6)

Figure II-6: HANNA conductivity meter EC 215

10. Turbidity measurement

Turbidity is the measurement of water's cloudiness, the opposite of clarity. Technically, turbidity is the optical property of water that allows incident light to be deflected (diffraction) or absorbed by particles rather than transmitted in a straight line. The turbidimeter used is AQUALYTIC (figure II-7)

Figure II-7: Turbidimeter type AQUALYTIC

11. Dry matter content Determination (DM)

Dry matter content is the percentage of solid material remaining in a substance after removing all water and moisture. It is a critical parameter in many applications because it indicates the concentration of solids or nutrients present in the material.

Procedure (figure II-8)

Fill a becher with a 60 ml sample of the material.

Measure the initial weight of the sample, put it in an oven set at a specific temperature around 105 C° for 24 hours and measure the new mass

Figure II-8: determination of Dry matter content

Dry matter content is expressed as a percentage of sample weight.

DM (%) = W1-W2 ⁄ W1 *100……………. (4)

Such as:

W1: wet sample mass. (g)

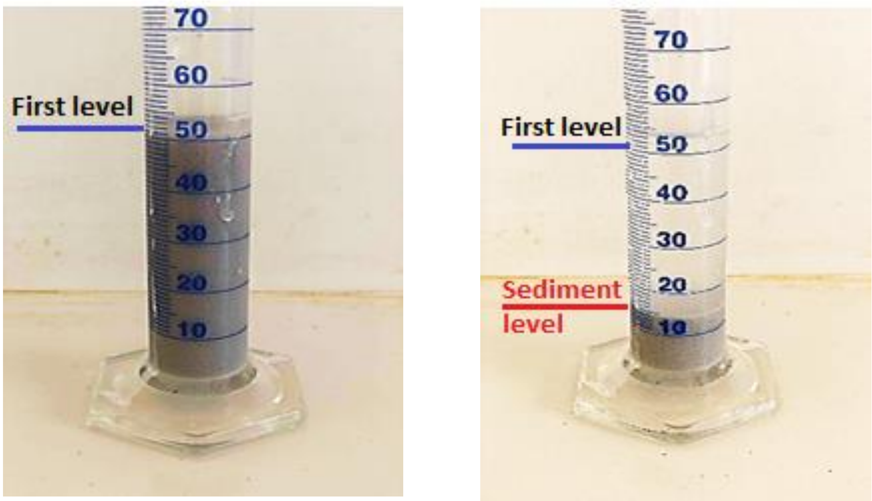
W2: mass of sample after steaming. (g)

12. Determination of Sedimentation rate

Sedimentation is a physical process whereby solid particles settle to the bottom of a liquid under the influence of gravity. The water is left to stand for a period of time, allowing heavier particles to settle to the bottom

Procedure

53ml of homogeneous liquid is transferred into a 100ml test tube. The height of the water-mud interface is noted as a function of time (figure II-9).



Sample at initial time **Sample sedimentation after time t**

Figure II-9: Sedimentation analysis

13. Sulfate level [5]

By measuring the barium sulfate ($BaSO_4$) precipitate obtained by reaction with barium chloride ($BaCl_2$), the sulfate content is evaluated in terms of gypsum ($CaSO_4$, $2H_2O$). $BaCl_2$ $^{+}$ $CaSO_4$ $BaSO_4$ $^{+}$

Procedure

1 g of the sample and 10 ml of 10% hydrochloric acid (HCl) are combined in a beaker. On a heated plate, the beaker is brought to a slow boil for ten minutes. The contents of the beaker are filtered into a flask after the boiling period has ended. To the filtrate, distilled water is added up to the 250cc mark. A beaker containing 100 milliliters of the filtrate is agitated vigorously. For ten minutes, the beaker is set on a heated pan. 10 ml of barium chloride ($BaCl_2$ 10%) is added to the contents of the beaker after the initial 8 minutes. The solution is filtered through paper once it has boiled. The contents of the filter paper are placed in a crucible for calcination in an oven at 900°C for 30 minutes.

Slowly boiling filtered solution Crucible before drying Crucible after drying

Figure II-10: Procedure of Sulfate level determination

Sulphate's content is expressed by:

Sulfate content = (P1 – P2)/P1 x 100……………. (5)

Where:

P1: Weight of crucible + filter paper. (g)

P2: Weight of crucible after furnace calcination. (g)

14. Determination of carbonates rate [6]

The carbonates test II-11 refers to a testing method used to determine the carbonate content in a sample. This test involves evaluating the carbonate content through a specific procedure, often using a calcimeter. The process typically includes measuring the volume of carbon dioxide released when the sample reacts with excess hydrochloric acid under known temperature and atmospheric pressure conditions. The results of this test provide information on the carbonate content of the material being analyzed.

Procedure

Accurately weigh 5 g of sample and place it in a beaker or Erlenmeyer flask. A known quantity of standard hydrochloric acid (HCl) solution is added to the sample. The obtained solution is mix while titrating with a standard solution of hydrochloric acid (HCl) from a graduated burette until a permanent colour change is observed. This colour change indicates the end of the reaction between the acid and the carbonates present in the sample.

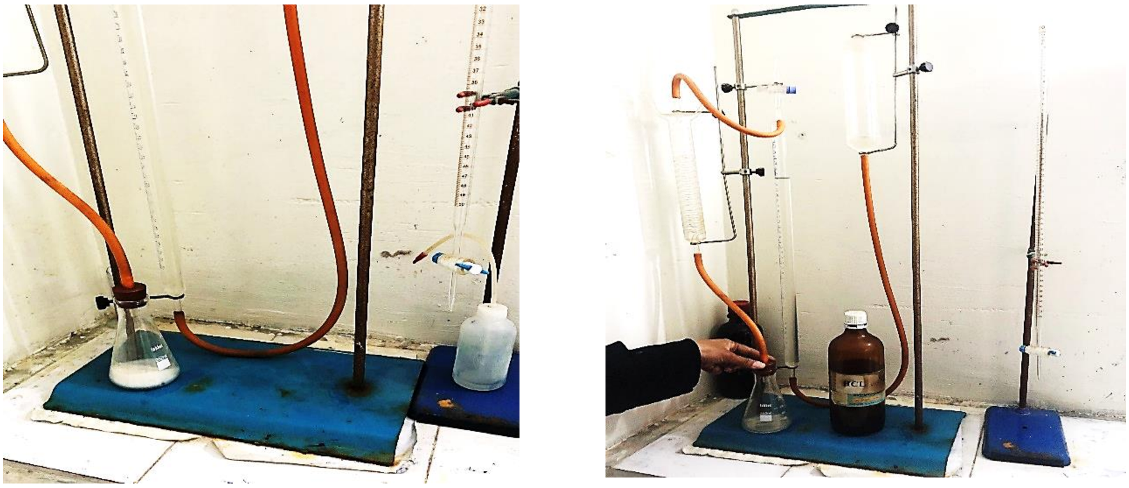


Figure II-11: Carbonates test

15. Density measurement

Density epitomizes the mass-to-volume ratio of a substance, calculated by dividing its mass by its volume. We can evaluate two density types: Density after compaction and apparent density.

15.1. Density after compaction [7]

Post-compaction density denotes the ratio of a material's mass to its volume subsequent to undergoing a specified compaction protocol. It quantifies the material's mass per unit volume following compression or compaction. Distinct from initial bulk density, post-compaction density is influenced by variables including compaction pressure, particle organization, and void spaces within the material matrix.

Procedure

In the density tester, a test tube (graduation cylinder) with 100g of materials is placed. The volume is read off after five minutes of tamping.

Figure II-12: Density tester JEL stampfvolumeter STAV 2003

Density after compaction is expressed by:

$$\textbf{Density(g/cm}^3\textbf{)} = \textbf{m/ V} \ldots\ldots\ldots\ldots\ldots \textbf{(6)}$$

Where

m: mass of the substance (g)

V: volume of the substance (cm^3)

15.2. Apparent density [8]

Apparent density, a fundamental material characteristic, quantifies the weight of an object or substance relative to its exterior volume adjusted for the volume of its open pores. It is determined by the ratio of the apparent volume to the dry mass of a specimen, accounting for external pressures and void spaces.

Procedure

Pour a quantity of material into a densimeter (apparent density funnel) until a pyramid is formed. Shave the surface of this pyramid with a straight edge, then weigh.

Densimeter (apparent density funnel) **Surface shaved with a straight edge**

Figure II-13: Determination of apparent density

The apparent density's calculi formula ρapp is as follows.

$$\rho app = \frac{M2 - M1}{V} \quad \text{.....................} \ (7)$$

Where

M2: the mass of container filled with leveled sample (g)

M1: the mass of empty container (g)

V: volume of the concainter (cm^3)

16. Determining slope angles: (Coulability)

Shear stresses can be transferred between particles in a powder pile composed of particles stacked on top of one another. If these interactions are insufficient to maintain the equilibrium of the particles, the pile collapses [9].

The management of granular media is initially based on the stability conditions and the statics of granular assemblies concerning the distribution of forces in an equilibrium state. For this, to study the equilibrium of a powder pile, the slope angle is determined.

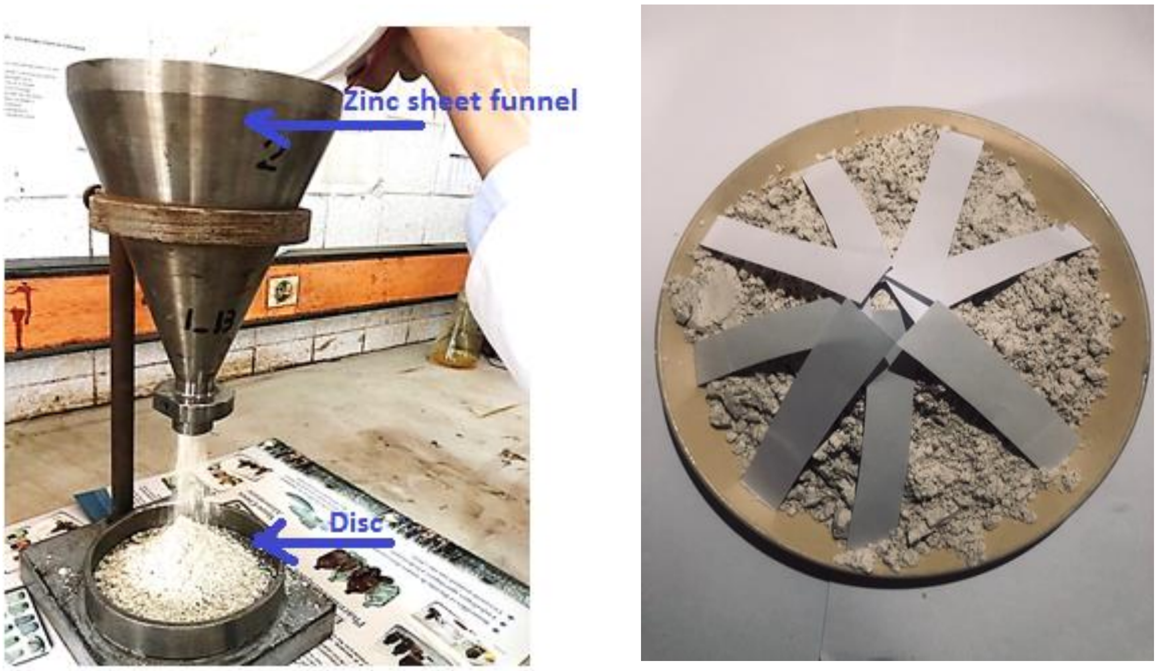
Procedure

In order to carry out the angle of slope measurements, it is necessary to suspend a zinc sheet funnel with a 20 mm diameter outlet above a 120 mm receiving disc.

It is necessary to determine the location of 08 measuring points to establish the minimum and maximum angles of slope.

The 20 mm diameter orifice of a funnel was positioned at 150 mm above the center of the receiving disc. The receiving disc is a ceramic plate with a diameter of 120 mm. (figure II-14 a)

In one go, 200 g of the sample is poured into the funnel and then poured onto the receiving disc. A pile is created as the sample moves.



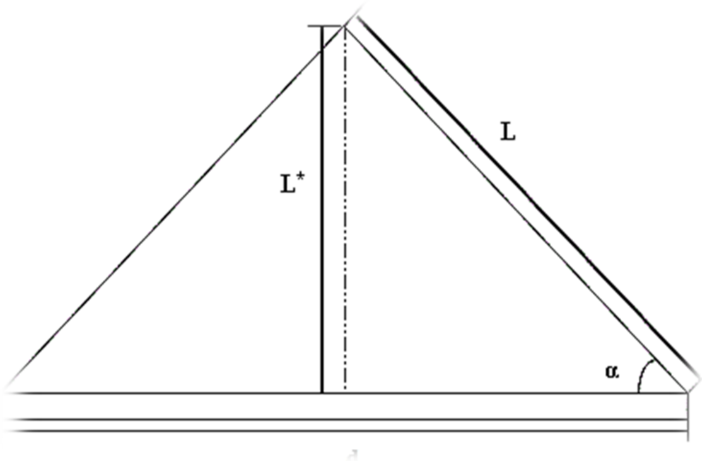
a) b)

Figure II-14: Determination of slope angle

The angle between the disc and the side of the heap formed by the material was measured at 08 points evenly distributed around the circumference of the disc (figure II-14 b).

The angle of repose was measured three times. The aim of this operation was to reduce the risk of error.

The angle of repose α is estimated as follows:



$$\text{Sin } \alpha = \frac{L^*}{L} \ldots\ldots\ldots\ldots\ldots \text{ (8)}$$

In which:

$$\alpha = \text{arc sin } \alpha \ldots\ldots\ldots\ldots\ldots \text{ (9)}$$

This property can be evaluated using two indices: the Hausner index and the Carr compressibility index.

- **The Hausner index (HI)** is determined by the following relationship:

$$IH = \frac{\rho r}{\rho app} \ldots\ldots\ldots\ldots\ldots \text{ (10)}$$

-The Carr index (CI) is evaluated according to the following relationship:

$$IC\ (\%) = \frac{\rho r - \rho app}{\rho app} \times 100 \ldots\ldots\ldots\ldots\ldots \text{ (11)}$$

Where:

ρr: real density in kg/m^3.

ρapp: apparent density in kg/m^3.

The Hausner index is used to classify the powder into the different categories shown in table 01

Table 01: Hausner compressibility index.

The Hausner index	Powder
IH≤1,25	*Sandy or granular*
1,25 < IH< 1,4	Flare
IH ≥ 1,4	*Cohesive*

The Carr Index (CI) % interprets the Coulability of the powder according to table 02

Table 02: Powder Coulability.

IC (%)	Flowability	Powder
5 to10	Excellent	*Granules, sands, powder without fine particles or fibers*
10 to15	Good	
15 to 25	Mediocre	*Powder with low particulate matter and high density*
25 to 30	Bad	*Powder containing fine particles*
30 to 40	Very bad	*Cohesive powder*
>40	Execrable Powder	*Highly cohesive*

16.1. Evaluation of angle of motion (α_m):

The flowability of a powder is its ability to flow freely, evenly and consistently in the form of individual particles.

The angle of movement is calculated as the average of the maximum values of each test over the angle of repose.

Procedure:

The angle of movement is the average of the maximum values obtained for the 03 tests, estimated as follows:

$$\alpha_m = \frac{(maxtest1) + (maxtest2) + (maxtest3)}{3} \quad \text{................ (12)}$$

16.2. Evolution of angle of repose (αr):

The angle of repose forms a constant angle with the horizontal of around 30°. This angle reveals the existence of frictional forces, responsible for the natural slope of the heap. The value of the angle of repose is the average of the values measured on different slope angle tests.

Procedure:

The angle of repose is the average of the mean values obtained for the 03 tests, estimated as follows:

$$\alpha_r = \frac{(moytest1) + (moytest2) + (moytest3)}{3} \quad \text{................ (13)}$$

17. Determining dispersibility

The dispersibility of the powder is characterized by its behavior as it falls through the air and its tendency to naturally scatter in the atmosphere.

Procedure

Dispersibility is assessed using a simple set-up. Its structure consists of a fixed cylinder 130 mm long and 50 mm in diameter, located 200 mm above a watch glass 120 mm in diameter. A 10-g sample is dropped in mass through the cylinder above the glass (figure II-15). The material collected in the watch glass is measured. Dispersibility is a direct assessment of the material's ability to move in a fluid manner.

To check the reproducibility of the measurement, the test was repeated three times.

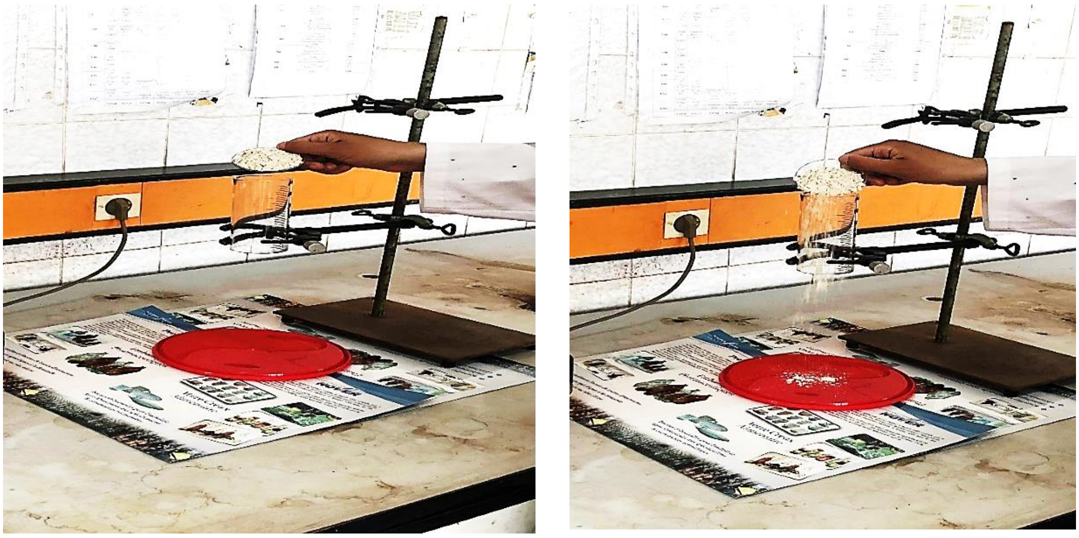


Figure II-15: Determination of dispersibility

The dispersibility D is calculated as follows:

$$D=\frac{m1-m2}{m1}\times 100 \quad (14)$$

Where

- m1: mass of sample (g) before dispersion.

- m2: mass of sample (g) dispersed.

18. Material slurry characterization

Sludges are generated using the materials investigated for their application in the polishing process. The purpose of the following tests is to determine whether the components used influence the consistency of the slurry.

18.1. Slurry conception

Mud samples were made by combining distilled water with specified amounts of substances following a ratio of 1 g sample powder (desired granulometry) to 5 mL distilled water.

19. Porosity

Porosity is a measure of the void spaces (pores) within a material. It is expressed as a fraction or percentage of the void volume and the total volume of the material.

***Procedure* [10]**

To characterize the porosity of a sample, its mass is measured and placed it in a volumetric flask, noting the apparent volume (Vapp). Add methanol (Vm) until the sample is completely submerged, indicating the volume of voids. Calculate the porosity using the formula

$$\textbf{Porosity (\%)} = V_m / V_{app} \cdot 100 \quad (15)$$

References

[1] Standard Methods Committee of the American Society for Testing and Materials (2008). D7348-08e1 Loss on Ignition (LOI) In: Annual Book of ASTM Standards. ASTM International, editors. West Conshohocken, PA: ASTM International.

[2] Standard Methods Committee of the International Organization for Standardization. ISO 13317-2:2019 Granulometric analysis (method: dry after washing) In: ISO Standards. ISO, editors. Geneva, Switzerland: ISO.

[3] Standard Methods Committee of the International Organization for Standardization. NF P 94-057 (1992) Granulometric analysis by sedimentation In: AFNOR Standards. AFNOR, editors. La Plaine Saint-Denis, France: AFNOR.

[4] Standard Methods Committee of the American Public Health Association, American Water Works Association, and Water Environment Federation. 2510 conductivity In: Standard Methods for the Examination of Water and Wastewater. 2023 Lipps WC, Baxter TE, Braun-Howland E, editors. Washington DC: APHA Press.

[5] ASTM C1580-15. (2015b). Standard test method for water-soluble sulfate in soil. ASTM International, West Conshohocken, PA.

[6] Determination of carbonates rate Standard Methods Committee of the International Organization for Standardization. ISO 13317-2:2019 Determination of carbonates rate In: ISO Standards. ISO, editors. Geneva, Switzerland: ISO.

[7] Standard Methods Committee of the International Organization for Standardization. ISO 527-3:2018 Density after Compaction In: ISO Standards. ISO, editors. Geneva, Switzerland: ISO.

[8] Standard Methods Committee of the International Organization for Standardization. ISO 60:2017 Apparent Density In: ISO Standards. ISO, editors. Geneva, Switzerland: ISO.

[9] Lubert, M. *Aptitude à l'écoulement d'un milieu granulaire; exploitation des instabilités de cisaillement,* thèse de doctorat, académie d'Aix-Marseille1, 2000.

[10] Zhang, R., & Ma, P. X. (1999). Poly (α-hydroxyl acids)/hydroxyapatite porous composites for bone-tissue engineering. I. Preparation and morphology. *Journal of Biomedical Materials Research: An Official Journal of The Society for Biomaterials, The Japanese Society for Biomaterials, and The Australian Society for Biomaterials*, *44*(4), 446-455.

CHAPTER III

MATERIALS CHARACTERIZATION

1. Introduction

This chapter presents the results and discussions of the raw materials used to the research objectives. The primary objective is to develop a low-cost abrasive material.

The physical characteristics of the raw materials in terms of their composition; particle size and surface area were established. As abrasive material is generally used in slurry form when polishing removable dental appliances, the materials particle suspension properties of the used materials are investigated. The functional groups were identified by Fourier transform infrared spectrometer IRTF. This study's reference was a marketed product that was identified as pumice.

All preparation and development of the low cost abrasive material was done at the Engineering process Laboratory (Moulay Taher University). The control quality laboratory of cement plant Hassassna. assisted in ball milling and characterising the materials. The most Physical properties were determined at Abras Saida laboratory.

2. Purchased Abrasive material (commercial product)

For the purpose of this investigation, a commercially available product named Pumice was characterized and subsequently compared with other available polishing agents.

2.1. Origin and pre-treatment of the commercial product

In this study, Pumice produced by Hakka dental group (figure III-I), was used as reference of abrasive material for polishing process. This item was purchased from a Dental Store Equipment in Saida city. Before analysis, the matter was mixed by a spatula

Figure III-1: Purchased Market product

The physical aspect of the market product is given in table 1.

Table 1: Physical aspect of purchased Abrasive Material

Material	Colour	odour	state at room temperature
Market product	Beige	Faint smells	solid

3. Chemical composition

Chemical composition for the market product is exposed in table 2

Table 2: Chemical composition of the market product

Oxide	CaO	Fe_2O_3	SiO_2	Al_2O_3	MgO	K_2O	SO_3	Na_2O	LOI
Market product (wt%)	23.472	1.265	49.41	2.71	5.656	0.716	0.31	1.293	12.47

The chemical analysis of the pumice (marketed product) as seen from the values show that the two most significant components of the sample are SiO_2 and CaO with the average amounts of 49.41% and 23.47 %, respectively. Moreover, an iron oxide content (1.27%) is observed. It can be concluded that the product isn't a pure pumice. The higher the silica percentage, the purer the pumice will be [1]. Other oxides have also been detected, such as MgO, Na_2O and K_2O. The LOI value of the marketed product is so higher.

The pH of market product suspension is alkaline. The sulphate is present in the product with a rate of 2.1% (table3). The rate of moisture is elevated.

Table 3: Chemical properties of the market product

Property	Humidity (%)	Sulphates (%)	$CaCO_3$ (%)	pH
Market product	9	2.1	45	8.89

3.1. Analysis by stereo microscope

Under the stereo microscope, the commercial polishing agent appears as macroscopic entities with a matte white appearance. It presents itself in an agglomerated and displays an opaque gloss under such magnification. The grain morphology of the sample is difficult to discern with a binocular microscop.

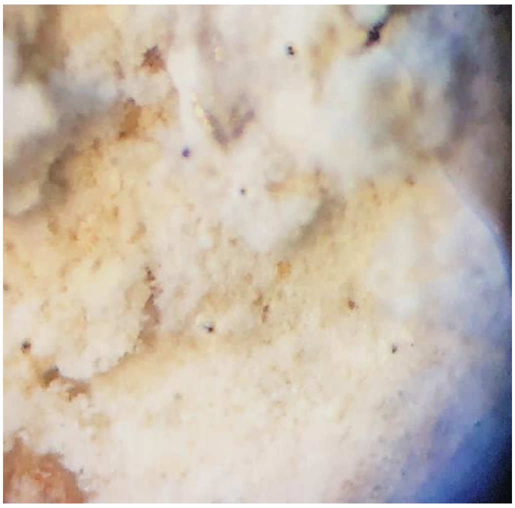


Figure III-2: Stereo microscope analysis of commercial polishing agent

3.2. Granulometry analysis

Both the wet and dry process particle sizes are shown for the particle size distributions.

3.2.1. Granulometric analysis by sieving (dry method)

Table 10 presents the market product particle size distribution.

Table 4: Granulometric analysis of the market product

Sieve diameter (mm)	Refus (%)
5.00	0
2.00	2.44
1.00	7.96
0.40	20.47
0.20	31.84
0.10	45.23
0.080	45.53

The commercial product has particles less than 5 mm.

3.2.2. Granulometric analysis by sedimentometry

Table III-5 presents the particle size distribution of the commercial product by sedimentation.

Based on the collected results, we draw a particle size curve that represented the weight-weight cumulative percentages of grains that passed through each consecutive sieve, in the sample under study. The obtained grain size curve (figure III-3) indicates that a market product material comprising 50% limon (0.02µm) makes up the majority of the polishing agent used. The findings show that the polishing agent, currently on the market, also has a gravel percentage of

1% (5µm) and 9 % (2µm) coarse sand. Additionally, there is an estimated 20% fine sand percentage (0.2µm) in the product.

Table 5: Granulometric analysis by sedimentometry of the market product

Time	Density	Corrected Density Ct	W% on sieve diameter <80µm	sieve diameter (µm)
15sec	20	20	84.82	80.00
30sec	19	19	80.58	58.00
1min	18	18	76.34	55.00
2min	17.50	17.50	74.22	38.00
5min	17	17	72.10	25.00
10min	15.50	15.50	65.74	17.00
20min	14.50	14.50	61	12.00
40min	13	13	55.13	8.00
80min	11	11	46.65	5.00
160min	08.50	08.50	36.05	4.00
320min	07	07	29.69	3.00
24h	04.50	04.50	19.08	2.00

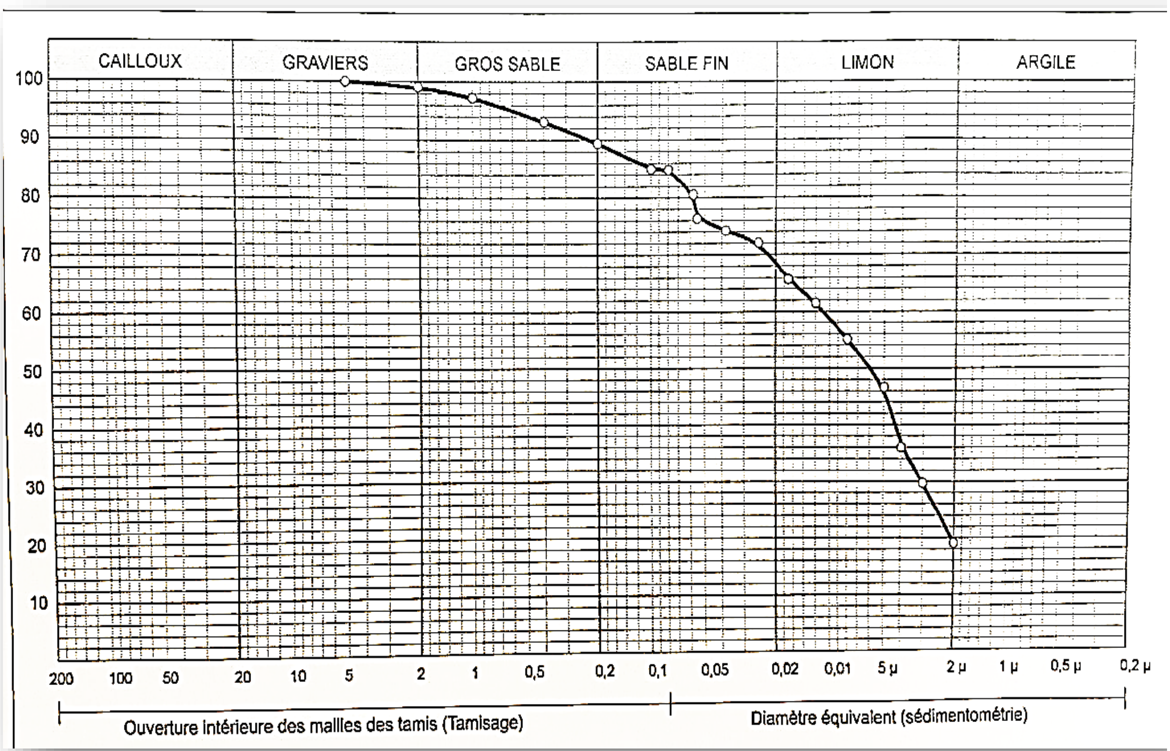


Figure III-3: The market product's granulometric curve

3.3. FTIR Identification

Figure IV shows the FTIR spectra of commercial product.

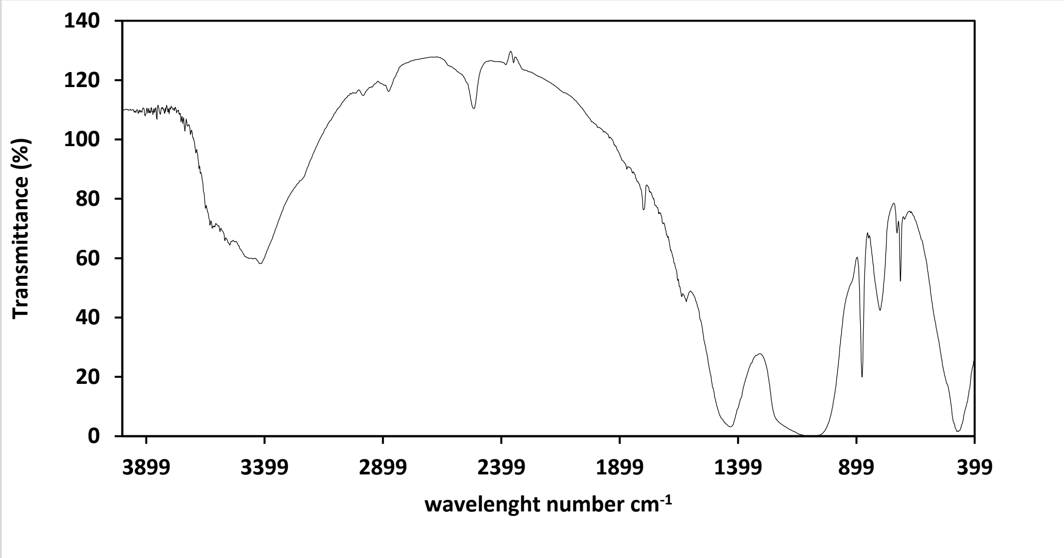


Figure III-4: FTIR spectrum of commercial product.

Figure IV shows the FTIR spectra of commercial sample. The peaks at 400 cm^{-1} in the FTIR spectrum of the commercial product may have resulted from the Si–O bending strength vibrations of the SiO_2, which constituted the structure. The absorption at around 1000 cm^{-1} corresponded to the Si-O-Si stretching vibration [2]. The other important peak was detected around 3413 cm^{-1}. This peak pointed out the OH stretching vibrations of the water (moisture) which was adsorbed by the sample from the outside environment [3]. While the peak around 1100 cm^{-1} may have resulted from Si–O stretching vibrations.

A shoulder at 875 cm^{-1} accompanied by a band at 1434.9 cm^{-1} is characteristic of C-O bond stretching vibration modes. These absorption bands highlight the presence of calcium carbonate [**4**].

3.4. XRD market product identification

Mineralogy of the commercial product was evaluated by XRD. The figure (Figure III-5) shows that the market product contains crystalline minerals which determinates that it is composed by calcite with peaks at 2Ɵ: 29.3°, 39.34°, 43°, 48.5° (3.04Å, 2.28Å, 2.09Å, 1.87Å). It identified also quartz peaks: 20.7°, 26.5°and 50.6° (4.28 Å, 3.36 Å, and 1.82 Å)

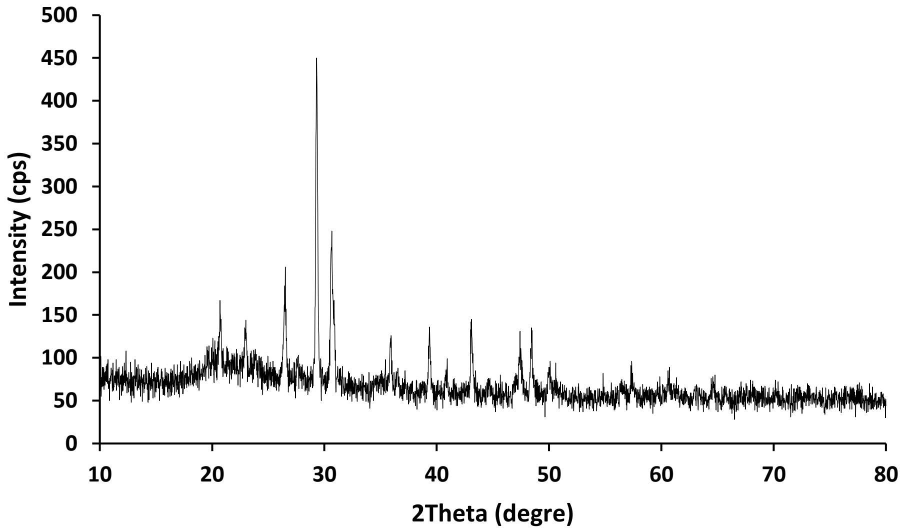


Figure III-5: XRD pattern of commercial product

3.5. Dry Commercial powder properties

The physical properties of the commercial product powder, including bulk density, moisture content, particle size and distribution, and flow characteristics, were investigated.

Grain pile densitometric characteristics may impact material processes including mixing and flow.

The different measured densities of the commercial product are shown in Table 6.

Table 6: measured densities of the market product

Property	Real Density (g/cm^3)	Apparent density (g/cm^3)	Tapped density (g/cm^3)	Porosity (%)
Market product	1.818	1.26	0.58	3.16

The commercial product is low dense. The Tapped density of the market product is similar to those of calcium hydroxide (0.40-0.58) [5].

Also measurements of slope angles were evaluated (table 7).

Table 7: Slope angle (°) measurement of commercial product powder.

		1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*Moy*	*Max*
commercial product	**Essay 1**	50.35	47.16	47.16	**50.35**	44.94	50.35	54.14	45.47	**50.35**	**54.14**
	Essay 2	48.82	49.54	57.49	51.05	58.61	44.99	46.19	51.86	**51.86**	**58.61**

Essay 3	43.47	48.59	47.33	53.75	47.95	49.93	45.58	47.33	**49.93**	**53.75**

The results of Table 3 are used to determine the commercial product's flowability (table 8)

Table 8: Flowability parameters of commercial product powder.

Flowability Parameters	**Slope Angles (°)**		**Hausner Indice**	**Carr index (%)**
	Motion Angle α_m	**Angle of repose α_r**		
Commercial product powder	55.5	50.71	1.44	44.28

The commercial product demonstrates substantial cohesion and very poor flowability, as indicated by an angle of repose exceeding 45°.

Table 9: dispersibility of the market product

	Dispersibility (%)		
Essay Number	**Essay 01**	**Essay 02**	**Essay 03**
Market	4.2	1.9	3.9

The dispersibility value (Figure 9) is challenging to reproduce accurately.

3.6. Market product slurry properties

Table 10 shows the properties evolution of the market product suspension.

Table 10: evolution of the market product slurry

Time	**0**	**1/2h**	**1h**	**3/2h**	**2h**	**5/2h**
pH	8.89	8.95	9.45	9.16	9.08	9.10
σ (mS)	1.80	2.06	3.12	2.06	1.44	1.60

The market product suspension's pH (table 10) gradually rises from 8.89 to 9.16 and stabilizes at 9.10. Additionally, the conductivity slurry grew over time and reached 1.6 mS of stability.

3.7. Conclusion

The market product composition shows that the product can't identify as a pumice which the CaO rate is down than 10%. The humidity percent revealed that this material is hydrophilic. The flowability character of the product is so bad. Its granulometry is less than 80 µm. The product slurry pH is 9.1

4. Industrial by-product

The industrial by product used in this study was obtained from Linde Gas (Sidi Belabess). It's a residue from Acetylene gas production. It is a carbide lime residue. Composed from calcium carbonate and calcium hydroxide, it was purposed ass polishing agent in this study.

4.1. Origin and pre-treatment of the industrial by product

Linde Gaz Algeria is situated 91 kilometers distant from Saida in the Sidi Bel Abbes industrial zone. This manufacture produces 26000 m^3 of acetylene/year annually.

Approximately 90 tons of carbide lime as residue of lime milk treatment, are generated annually, where is discarded in EPIC of Sidi Belabes

The lime milk sample was collected by an operator on 24/01/2024 at the settling pit and it was stored in a five Liter drum. While the carbide lime residue from stockpile of acetylene gas unity was brought to laboratory in plastic bags.

After we gather the sample, we place it under the sun to dry for a week and then we put it in the oven till it get dried at T = 105°C. After that we grinded it until it become a fine powder.

For the carbide lime milk, we put it in a basin and let it sit at ambient temperature (figure III-6).

industrial by-product collected | **industrial by product after treatment** | **carbide lime milk sedimentation**

Figure III-6: Industrial by-product samples

The samples' physical aspect is given in table 11

Table 11: Industrial by product aspect

Material	**Colour**	**odour**	**state at room temperature**
lime milk	Grey	strong smell	liquid
Carbide lime	Grey	Faint smell	Solid

4.2. Lime milk properties

The lime milk sample properties were evaluated in order to to take use of it.

The pH evolution over time of the lime milk is shown in table 12

Table 12: pH evolution of lime milk

Time (h)	0	1/2h	1h	2h	3h	24h
pH	12.38	12.10	12.24	12.36	12.42	12.29

The conductivity decreases steadily with increasing sedimentation time (table 13). Similarly the solids dissolved concentration decreases.

Table 13: Conductivity evolution of lime milk

Time	0	½	1	2	3
σ (mS)	22.9	17.01	10.4	7.6	9.2

Other properties of milk of lime were estimated to predict future uses (figure 14).

Table 14: Chemical properties of the lime milk

Property	**Dry matter rate (%)**	**Turbidity (NTU)**
Lime milk	78.54	1931

The Sedimentary lime milk kinetics are given in Table 15

Table 15: Sedimentary lime milk kinetics

Time (min)	0	4	8	10	14	24
Sediment volume (ml)	53	25	18	16	15	14

After twenty-five minutes, the lime milk is completely decanted (table 15). This period is necessary to recover the residue (figure III-7), which consists of hydrated lime, in order to repurpose it for the development of the novel material that is the focus of this investigation.

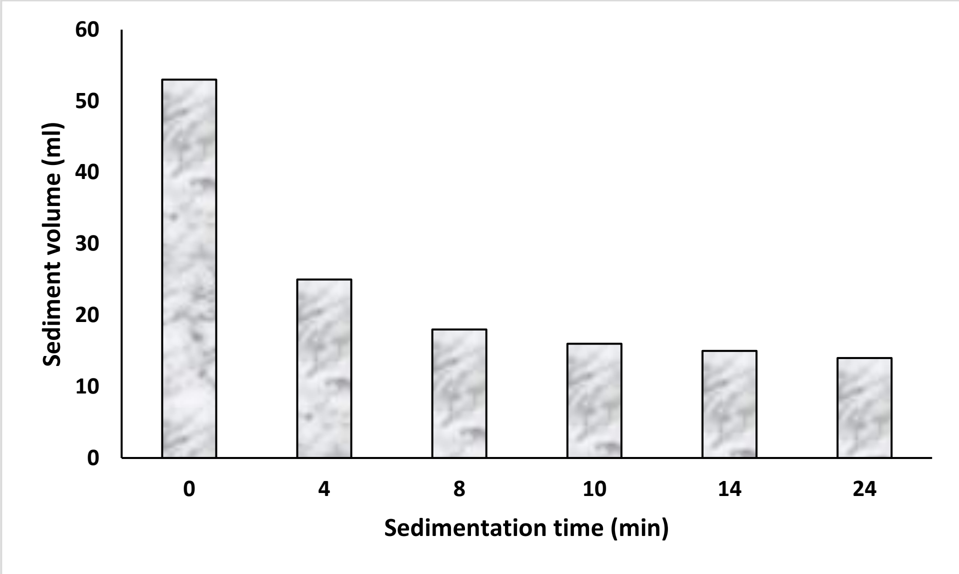


Figure III-7: Evolution of lime milk sedimentation

4.3. Carbide lime characterization

The solid waste of Linde Gas was characterized to valorize it as polishing agent.

5. Chemical composition

Results of the material components evaluated by X-ray Fluorescence spectrometry are shown in Table 16

Table 16: Chemical composition of by product (Wt. %).

Oxide	CaO	Fe_2O_3	SiO_2	Al_2O_3	MgO	K_2O	Na_2O	SO_3	LOI
Industrial by-product (wt%)	65.98	0.13	2.75	0.96	0.21	0.27	0.035	0.733	29.41

The results of the chemical analysis of the by-product indicate that CaO, which had an average level of 65.98%, was the most significant component of the sample. Additionally, a notable SiO_2 concentration (2.75%) is noted, which is insufficient when compared to the product on the market. There have also been discoveries of other oxides, including MgO, Na_2O, CaO, and K_2O. The loss of volatile components like water or carbon dioxide is the reason for the product's extremely high LOI score of 29.41%.

With the exception of the 2015 study composition [6], when we felt that the selected etalon wasn't compatible with the lime composition, the Linde Gaz by Product shows a rather stable chemical composition over time (table 17).

Table 17: Chemical composition of carbid lime (Wt. %) (2014-2023) [6-10].

Raw Material	CaO	Fe_2O_3	SiO_2	Al_2O_3	MgO	K_2O	SO_3	Na_2O	LOI
2014 [7]	71.67	1.12	0.00	0.51	0.00	0.00	0.19	0.121	25.22
2015 [6]	58.20	0.24	5.17	1.90	1.44	0.17	/	/	30.35
2016 [8]	71.80	0.56	1.82	1.44	0.85	/	/	/	24.49
2019 [9]	74.303	0.135	2.836	1.42	0.109	0.28	/	/	17.76
2023 [10]	74.04	0.245	0.887	1.08	0.53	0.275	0.887	0.447	20.81

The by-product is still wet in basis of its humidity (table 18)

Table 18: Chemical properties of the by product

property	Humidity (%)	Sulfates (%)	$CaCO_3$ (%)	pH	Porosity (%)
By product	27.84	1.634	27	12.60	3.71

5.1. Analysis by stereo microscope of the industrial by-product

The stereo microscope image of the industrial by-product is shown in figure VIII

Figure III-8: Stereo microscope analysis of industrial by-product

The fine particles of the finest sample appear in the form of agglomerates. This may indicate a cohesive behavior of these particles.

5.2. Granulometric analysis:

The particle size distribution of the carbide lime was performed.

5.2.1. Granulometry and sedimentometry analysis

The results of the hydrated lime particle size are given on granulometric analysis by dry method and sedimentometry.

5.2.2. Granulometric analysis by sieving (dry method)

Table 20 presents the granulometric analysis of industrial by-product by dry method.

Table 19: By-Product Granulometric analysis

Sieve diameter (mm)	Refus (%)
5,00	0
2,00	0.13
1,00	0.61
0,400	2.91
0,200	11.25
0,100	23.25
0,080	23.51

It is observed that over 50% of the hydrated lime grains have a diameter less than 5mm.

5.2.3. Granulometric analysis by sedimentometry

Table 20: By-Product Granulometric analysis by sedimentometry

Time	Density	Corrected Density Ct	P% on sieve diameter <80µm	Sieve diameter (µm)
15sec	22.00	22.00	92.16	80.00
30sec	21.00	21.00	87.97	58.00
1min	20.50	20.50	85.88	55.00
2min	19.50	19.50	81.69	38.00
5min	16.50	16.50	69.12	25.00
10min	10.00	10.00	41.89	17.00
20min	0	0	0	0
40min	0	0	0	0
80min	0	0	0	0
160min	0	0	0	0
320min	0	0	0	0
24h	0	0	0	0

From the results obtained, we plotted the particle size distribution curve, representing the cumulative weight percentages of grains passing through the successive sieves of the industrial by-product. The grain size curve (figure III-9) reveals that the by-product used is predominantly a mineral material, comprising 92.16% of size grains 80µm. The findings indicate that the carbide lime brickworks also contains a limon fraction (0.02µm) of 9 % and 2 % of coarse sand (2µm) along with a fine sand fraction (0.2µm) estimated at 48%.

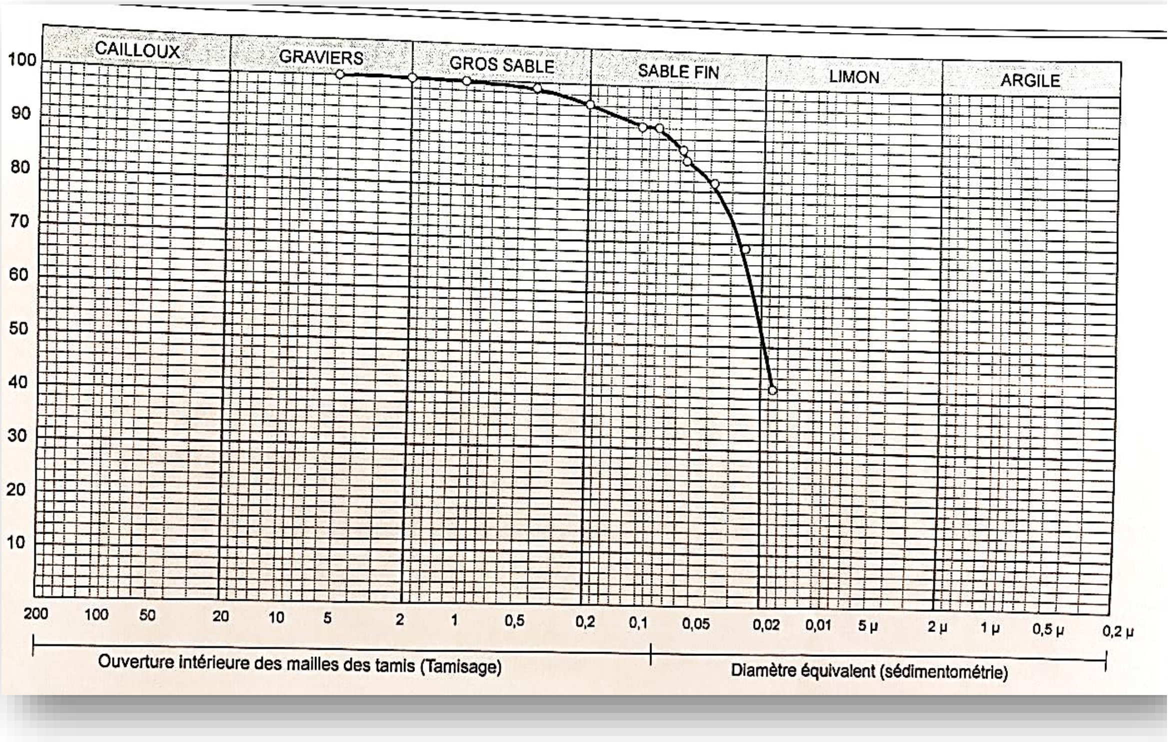


Figure III-9: The carbide lime granulometric curve

5.3. FTIR Characterization

Figure III-10 shows the FTIR spectrum of industrial by-product

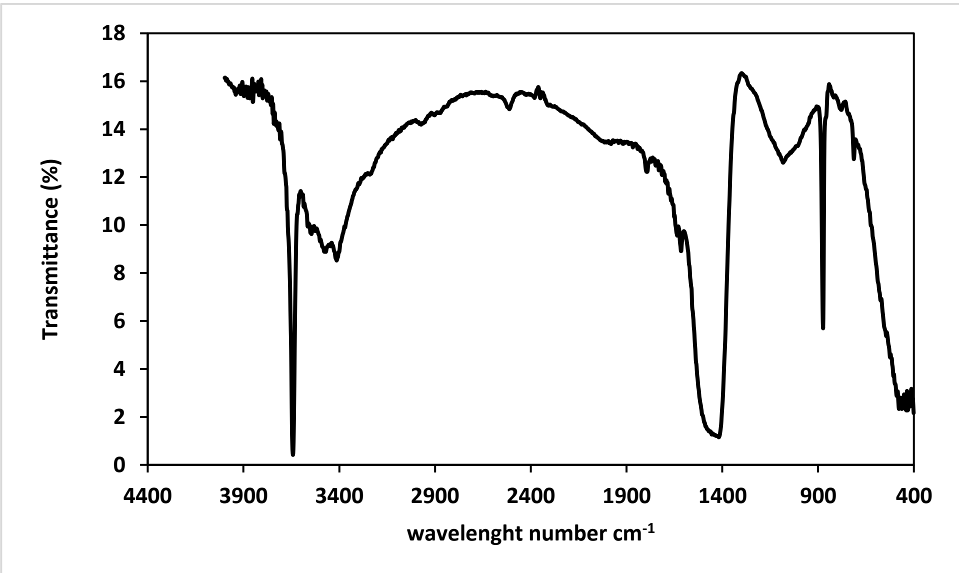


Figure III-10: FTIR spectrum of carbide lime

The results of FTIR show that this material tends to report broad band centred at 1423 cm^{-1}, which is attributed to the lime band present in calcium carbonate [11].

FTIR analyse (Figure III-10) shows the characteristic band of the H–O–H bending vibration at 1629 cm^{-1} and the strong O–H stretching band at 3640 cm^{-1} are due to the presence of $Ca(OH)_2$ which is only present in lime hydrate [12]. The small sharp peak at 881 cm^{-1} corresponds to the asymmetric deformation of CO_3 groups. The band at 1076 cm^{-1} of calcite is observed [13]. The Band observed at around 3400 cm^{-1} is due to vibrations of hydroxyl groups.

5.4. XRD identification carbide lime

X-ray diffraction pattern of carbide lime is shown in figure III-11.

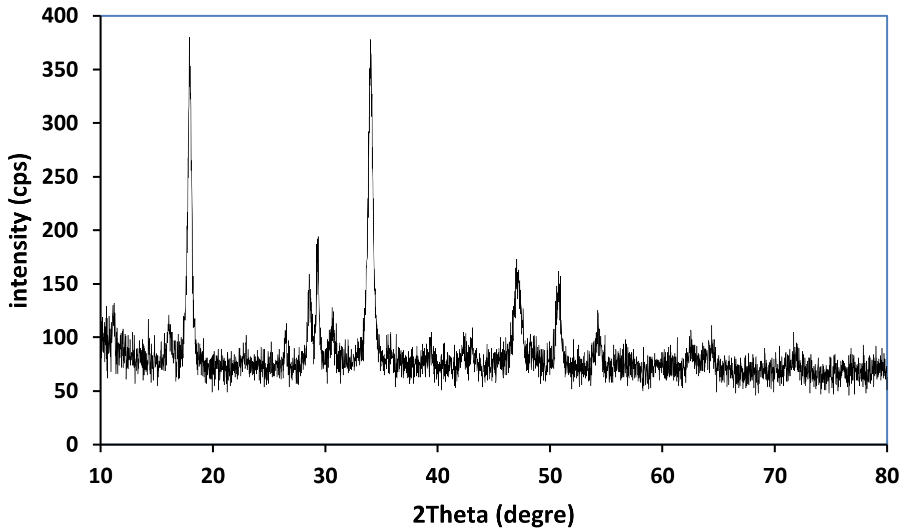


Figure III-11: XRD pattern of carbide lime

These results show the presence, as dominant minerals, of portlandite with peaks at 2theta position: 34°, 17.21°, 28.6°, 50.84°, 54.55° (2.63 Å, 4.94 Å, 3.12 Å, 1.8 Å, 1.68 Å), and calcite at: 29.42°, 46.96° (3.04 Å, 1.93 Å). It also identified carbon as graphite in the by-product at 26.46° (3.35 Å). Other researchers [14 -15] also reported these results

5.5. Industrial by-product powder properties

The density of eggshell powder was measured (table 21).

Table 21: Measured densities of the by product

Property	Real density (g/cm^3)	Apparent density (g/cm^3)	Tapped density (g/cm^3)	Porosity (%)
The industrial by product	2.22	1.545	0.98	3.71

The results show that the tapped density of the by-product is similar to that of the calcium carbonate (0.96-1.1) [5]. Otherwise the real density's by-product is closer to hydroxide calcium's (2.7g/cm3). The same result was reported by Pelisser and al. [16]

Density results are probably related to the chemical composition as the industrial residue has a smaller content of carbonate, which is denser than the hydroxide (2.71 and 2.26 g/cm3, respectively). The presence of a minor portion of carbon, which density can vary from 1.8 to 2.25 g/cm3, depending on the crystallinity, also favors the lower density of CL. [17]. The by-product has a high packed density due to its porosity.

The angle of slope is a relatively simple measurement that reflects the static or restricted flow mechanism of powders, making it the most useful indicator of their physical behavior. Table 22 presents the slope angles of industrial by-product.

Table 22: Talus angle (°) measurement of industrial by-product powder.

		1	***2***	***3***	***4***	***5***	***6***	***7***	***8***	***Moy***	***Max***
industrial by-product	**Essay 1**	49.25	39.86	34.62	**41.81**	45.58	45.58	38.68	41.81	**41.81**	**49.25**
	Essay 2	51.05	52.21	53.44	51.05	57.65	56.15	53.44	**59.27**	**53.44**	**59.27**
	Essay 3	50.18	54.62	48.28	49.21	57.27	**54.62**	53.42	47.40	**49.21**	**54.62**

Measuring the slope angle (table 23) allows for the classification of powders according to their flow regime.

Table 23: Flowability parameters of industrial by-product.

Flowability Parameters	**Slope Angles (°)**		**Hausner Indice**	**Carr index (%)**
	Motion Angle α_m	**Angle of repose α_r**		
Industrial by-product powder	54.38	48.15	1.436	43.68

The angle of repose or the ability of the material to flow through orifices of varying diameters indicate that it exhibits significant cohesion.

Table 24 displays the results of the 3 tests used to evaluate the dispersibility index.

Table 24: Dispersibility of the industrial by-product

	Dispersibility (%)		
Essay Number	**Essay 01**	**Essay 02**	**Essay 03**
Industrial by-product	2.9	1.9	3.3

Since the particles do not scatter, the powder exhibits fluid flow.

5.6. By product slurry properties

Table 26 shows the properties of the by-product suspension

Table 25: Evolution of the by-product slurry properties

Time	0	1/2h	1h	3/2h	2h	5/2h
pH	12.45	12.66	12.70	12.72	12.58	12.81
σ (mS)	6.32	6.85	6.21	6.09	5.84	5.78

Table 26 shows the pH value of the by-product suspension As expected for Carbide Lime with high levels of $Ca(OH)_2$, the pH is close to 12. Its conductivity is average equal to 6mS.

5.7. Conclusion

The result of industrial waste analysis is promising to use it as polishing agent, based on the following conclusions. Carbide lime contains an appreciable amount of portlandite and calcite as mineral materials. The pH by-product suspension is 12.

An abrasive precipitated calcium carbonate is provided that provides excellent cleaning properties without being excessively abrasive or damaging to gums or tooth Surfaces. The precipitated carbide lime has a primary particle Size of about 80.µm. The calcium hydroxide slurry is maintaining constant pH.

6. Chicken Eggshell

In this study, polishing agents were synthesized using chicken eggshells.

6.1. Origin and pre-treatment of the Eggshell powder

Eggshells were first collected from household cooking waste over a period of three days. The following experimental protocol (figure III-12) was used to produce a polishing agent from raw eggshells.

250 g of eggshells are washed with water to remove any organic matter. Afterward, there were soaked again with a 10% diluted sodium hypochlorite solution.

Subsequently, eggshells were dried first for 6-9 minutes at 250°C, crushed to a powder with a grinder in order to increase the efficiency of mechanical reaction with larger surface areas, dried at 80°C for 2 hours, ground to a powder with a grinder, and sieved through a fine mesh.

Eggshell in sodium hypochlorite solution

Eggshell after being soaked in the solution

Eggshell dried in the oven

Grinder tool: the rings

Ring Grinder grinding eggshell

Eggshell powder

Figure III-12: Preparation of eggshell powder

Table 26 provides a summary of the product's appearance.

Table 26: Physical aspect of the eggshell

Material	colour	odour	state at room temperature
Egg shell powder	Beige	Strong smell	solid

7. Chemical Composition

The chemical composition of the eggshell powder was determined, and the analysis yielded the following results (Table 27).

Table 27: Chemical composition of the eggshell powder

Oxide	CaO	Fe_2O_3	SiO_2	Al_2O_3	MgO	K_2O	SO_3	Na_2O	LOI
Eggshell powder (wt%)	51.58	0.02	0.54	0.22	1.05	0.21	0.772	0.295	46.40

Eggshell powder is distinguished by its high CaO content (51.58%). It has the highest loss on fire (46.40%). This rate of loss on ignition is close to that of ulterior studies [18].

Table 28: Chemical properties of eggshell powder

Property	Humidity (%)	Sulfates (%)	$CaCO_3$ (%)	pH
Eggshell	0.01	1.495	86	9.70

As illustrated in Table 28, the eggshell powder had a natural moisture content of 0.01 % on average. The pH value of the Eggshell suspension is 9.70. The CaO is present as Calcium carbonate. This indicates that the material has a high level of carbonate. Sulphates are not a significant eggshell powder component. Using eggshell as a dental polishing agent is advantageous because its predominant inorganic mineral, calcium carbonate, is inherently a poor source of nutrients for microbial colonization. Consequently, it inhibits the colonization of bacteria [19].

7.1. Eggshell powder Analysis by stereo microscope

Figure III-13: Eggshell powder Analysis by stereo microscope

The stereo microscope image (Figure III-13) showed that the Eggshell particles are clustered together, thereby causing particle agglomeration.

The particles are brilliant.

7.2. Granulometry analysis

Eggshell powder granylometry is determinated following the dry method and by by sedimentometry

7.2.1. Granulometric analysis by sieving (dry method)

Table 29 presents the particle size distribution of the utilized Eggshell.

Table 29: Eggshell Granulometric analysis

Sieve diameter (mm)	Refus (%)
5,00	0
2,00	0
1,00	0,10
0,40	0,21
0,20	0,40
0,10	2,01
0,080	2,30

The particle size of Eggshell is less than 2mm.

7.2.2. Granulometric analysis by sedimentometry

Table 30 presents the particle size distribution of the Eggshell determined by sedimentation.

The sample is primarily composed of silty materials, as indicated by the particle size curve (figure III-14). Of it, 12% are fine silt (2µm). It is composed also of 56% fine sand (0.02µm).

Table 30: Eggshell Granulometric analysis by sedimentometry

Time	Density	Corrected Density Ct	P% on sieve diameter <80µm	sieve diameter (µm)
15sec	21	21	99.23	80.00
30sec	20	20	94.50	58.00
1min	20	20	94.50	55.00
2min	19	19	89.78	38.00
5min	16	16	75.60	25.00
10min	13	13	61.43	17.00
20min	12	12	56.70	12.00
40min	10	10	47.25	8.00
80min	09	09	42.53	5.00
160min	08.50	08.50	40.16	4.00
320min	06.00	06.00	28.35	3.00
24h	03.00	03.00	14.18	2.00

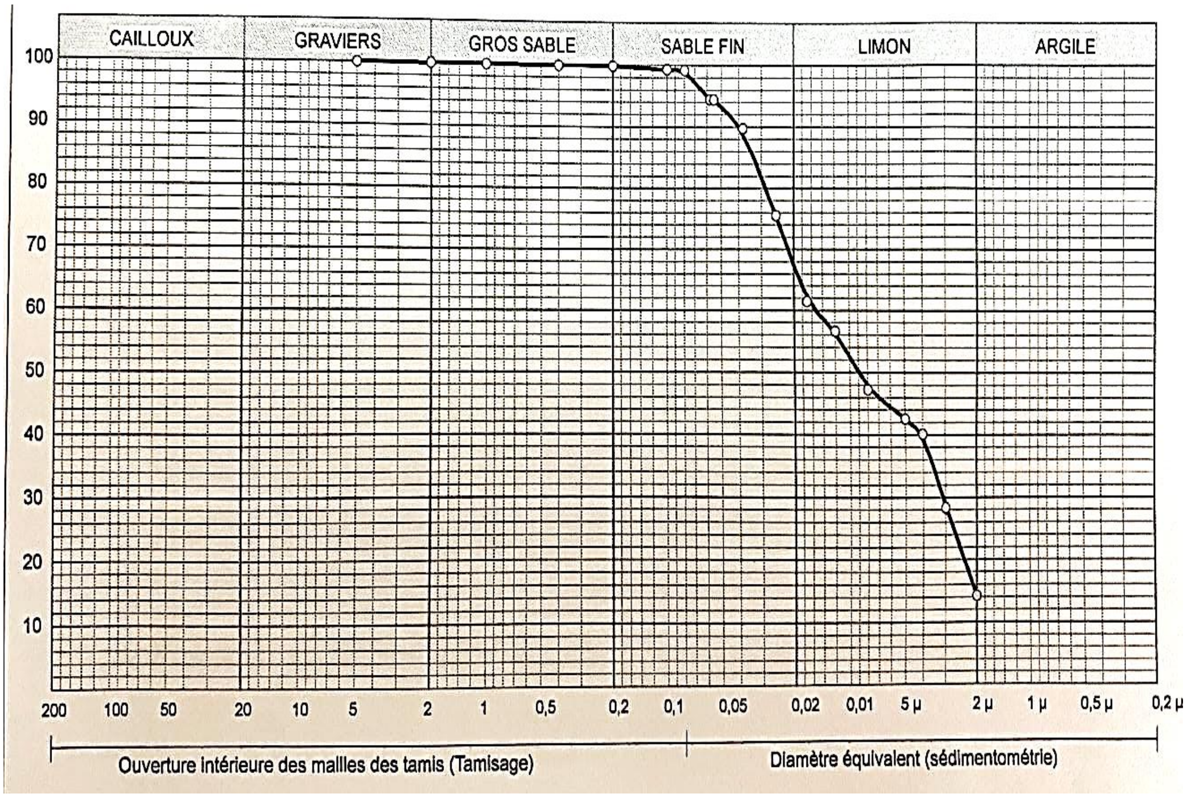


Figure III-14: Eggshell particle size curve

7.3. FTIR Characterization

The eggshell powder spectrum is shown in Figure III-15.

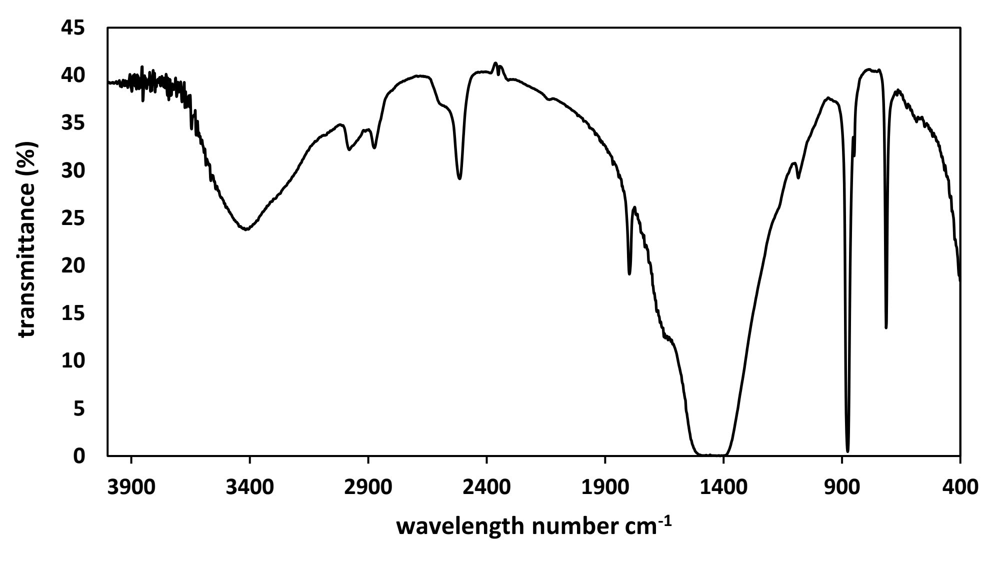


Figure III-15: Infrared spectra of eggshell powder

Figure III-15 demonstrates the infrared spectra of eggshell powder. The prominent absorption band associated with the stretching vibration of bond of O–C–O linkages is at 875 cm^{-1} [20]. Besides, the absorption band existing at 714 cm^{-1} can be assigned to the Ca–O bond [21, 22]. Furthermore, the two sharp absorption bands at 709 cm^{-1} and 871 cm^{-1}, which are correlated with out-plane and in-plane bending, respectively, indicate the existence of calcium carbonate ($CaCO_3$). The band occurring at 1076 cm^{-1} can be attributed to the vibrations of the carbonate $CO3^{-2}$ anions also the absorption band at 1405 cm^{-1} is attributed to asymmetric stretching vibration modes of CO_3^{-2} molecules [23, 24]. The broad band appears due to the presence of a C-O bond which reveals the bond between the oxygen atom of $CO_3{}^{2-}$ and the calcium atom. These findings indicate that the calcium oxides and carbonates were present and well-dispersed in the eggshell material as described by Busca and Resini [25]. Moreover, the weak IR peak at 2511cm^{-1} indicates the presence of organic matter in the Eggshell spectrum. With respect to the eggshell membranes, as expectable, the presence of amides were found, exhibiting significant peaks. The C-H vibration bands at 2978cm-1 and 2885 cm^{-1} confirm the presence of organic layers in the eggshells. The band at 2978 cm^{-1} represents C-H vibration, indicating the existence of the organic layers, built from amino acids, in the eggshells [26]. Band at 1795.6 cm^{-1} corresponds to C=O and carbonyl group stretching (amide) [27, 28].The band located at 3415 cm^{-1} in the spectrum has been attributed to H-bonded water (hydroxyl group stretching) due to humidity [29].

7.4. XRD identification

The Eggshell powder diffractogram is shown in the figure III-16

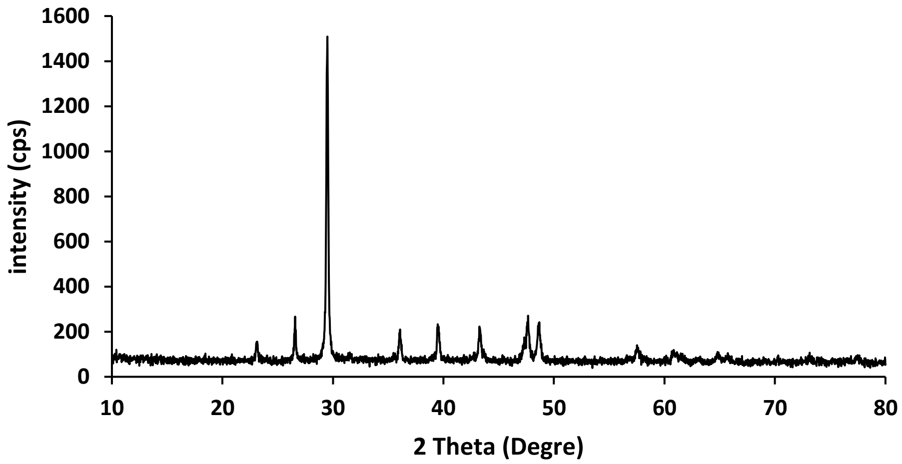


Figure III-16: XRD pattern of Eggshell

The observed peaks matched with fundamentals of optics by Sachinath Mitra [30], at peak position 29.46° (3.02 Å), 39.49° (2.28 Å), 43.26° (2.08 Å), 47.57° (1.9 Å), 48.57° (1.87 Å) ° and 57.55° (1.6 Å) matches and found that egg shell contains calcium carbonate as the major inorganic constituent. It is in the calcite form [31].

7.5. Eggshell dry powder properties

The density of eggshell powder was measured (table 31).

Table 31: Measured densities of the eggshell powder

Property	**Real density (g/cm^3)**	**Apparent density (g/cm^3)**	**Tapped density (g/cm^3)**	**Porosity (%)**
Egg shell	2.35	1.59	1.11	4

The detailed physical properties of the eggshell are as follows: The tapped density of eggshell is near that of calcium carbonate (0.96-1.1) [5]. The real density is near to the limestone ($2.7 g/cm^3$) [32]. The eggshell is the most porous material compared to the other polishing materials suggested in this study.

The slope angles were calculated to estimate the flowability of the powder. Table 32 presents the collected results.

Table 32: Slope angle (°) measurement of eggshell powder

		1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*Moy*	*Max*
EggShell	**Essay 1**	40.32	39.75	35.37	**39.21**	37.66	36.25	35.81	39.75	**39.21**	**40.32**
	Essay 2	46.05	46.86	44.53	49.51	45.27	44.53	48.59	**46.86**	**46.86**	**49.51**
	Essay 3	45.43	44.03	44.72	46.18	48.59	**45.43**	49.46	51.33	**45.43**	**51.33**

After calculation, the angles of motion and repose were estimated as presented in Table 33

Table 33: Flowability parameters of eggshell powder

Flowability Parameters	**Slope Angles (°)**		**Hausner Indice**	**Carr index (%)**
	Motion Angle α_m	**Angle of repose α_r**		
Eggshell Powder	47.05	43.83	1.477	47.79

According to Carr's classification [33], the flowability of the EggShell powder is good, indicating slight cohesion between particles (35° < αr < 45°). Eggshell powder is indicated by a compressibility index of Carr more than 45%, as highly coherent, with flowability that could only be described as very poor. Similarly, the Hausner index value of the eggshell powder exceeds 1.4, indicating that it is a cohesive powder.

Table 34 displays the results of the 3 tests used to evaluate the dispersibility index.

Table 34: Eggshell powder dispersibility

	Dispersibility (%)		
Essay Number	**Essay 01**	**Essay 02**	**Essay 03**
EggShell Powder	1.6	1.9	1.9

Since the particles do not scatter, the powder exhibits fluid flow.

7.6. Eggshell slurry properties

Table 35 shows the pH evolution of the egg shell suspension. The egg shell suspension is basic. The pH value is around 9. The egg shell slurry conductivity is almost 1.15mS.

Table 35: Evolution of the eggshell slurry

Time	0	1/2h	1h	3/2h	2h	5/2h
pH	9.25	9.03	9.12	8.92	8.97	8.96
σ (mS)	1.13	1.55	1.15	1.16	1.15	1.15

There were no variations of conductivity and pH in eggshell suspension which stated that there is no carbonation reaction.

7.7. Conclusion

Eggshell powder analysis revealed that this material has high levels of carbonate. The predominant inorganic mineral (calcium carbonate) is by its very nature a poor source of nutrients for microbes to colonise. The average mean size of the Eggshell measured 0.2.µm and 0.02.µm. The Eggshell particles are clustered together. The granulometry analysis reveals that powder particule size is fine. The powder has a bad coulability and is cohesive.

8. Conclusion

Phase One was premised on characterising an alternative abrasive material synthesised from eggshells and industrial by-product in order to ascertain their use as an abrasive material. Both FTIR and XRD analyses confirmed that the materials were composed by calcite with unique water absorbing characteristics. These results supported that materials could be free of bacteria. The enhanced physical and chemical characteristics of the samples confirm their application as an alternative abrasive material to pumice to polish removable dental appliances regarding their mineral composition and their granulometry.

The next tests part of our study is the research hypothesis by demonstrating the qualities of the proposed materials in reducing the surface roughness of area prothesis.

References:

[1] Ersoy, B., Sariisik, A., Dikmen, S., & Sariisik, G. (2010). Characterization of acidic pumice and determination of its electrokinetic properties in water. *Powder Technology*, 197(1-2), 129-135.

[2] Sepehr, M. N., Sivasankar, V., Zarrabi, M., & Kumar, M. S. (2013). Surface modification of pumice enhancing its fluoride adsorption capacity: An insight into kinetic and thermodynamic studies. *Chemical engineering journal*, 228, 192-204.

[3] Grim, R. E. (1968). *Clay Mineralogy*: 2nd Edn McGraw-Hill. New York.

[4] Tantawy, M. A., El-Roudi, A. M., Abdalla, E. M., & Abdelzaher, M. A. (2012). Evaluation of the pozzolanic activity of sewage sludge ash. *International Scholarly Research Notices.*

[5] National Lime Association. (2007). Lime terminology, standards & properties. Lime The Versatile Chemical Fact Sheet, Arlington, Virginia.

[6] Mehdi, A., Souiah, N. (2015) *Conception d'un ciment spécial à partir de déchets inertes* (Master's thesis, Dr. Moulay Tahar University Saida).

[7] Boutarane, H., Nekrouf, S. (2014) *Valorisation du lait de chaux résiduaire de l'entreprise nationale des gaz industriels -Sidi Belabess- dans l'industrie cimentière* (Licence's thesis, Dr. Moulay Tahar University Saida).

[8] Belarbi, O., Rezig, M., Nekrouf, S., & Marouf, R. (2017). Properties and potentialities of sewage sludge as a renewable energy source in cement manufacture. In *Proceedings of Engineering & Technology (PET)* (pp. 121-124). Tabarka, Tunisia.

[9] Bounecissa, K., Daoudi, K. (2019). *Traitement des eaux usées par coagulation– floculation en présence de matrices solides issues de résidus urbains* (Master's thesis, Dr. Moulay Tahar University Saida).

[10] Megharbi, I., Zougab, S. (2023). *Production d'un matériau de construction écologique* (Master's thesis, Dr. Moulay Tahar University Saida).

[11] Casarez, C. A., Arredondo-Rea, S. P., Gómez-Soberón, J. M., Alamaral-Sánchez, J. L., Corral-Higuera, R., Chinchillas-Chinchillas, M. D. J., & Acuña-Agüero, O. H. (2014). Experimental study of XRD, FTIR and TGA techniques in geopolymeric materials. Int. *J. Adv. Comput. Sci. Appl*, 4(4), 221-226.

[12] Cizer, Ö. Rodriguez-Navarro, C., Ruiz-Agudo, E., Elsen, J., Van Gemert, D., & Van Balen, K. (2012). Phase and morphology evolution of calcium carbonate precipitated by carbonation of hydrated lime. *Journal of Materials Science*, 47(16), 6151-6165.

[13] Addadi, L., Raz, S., & Weiner, S. (2003). Taking advantage of disorder: amorphous calcium carbonate and its roles in biomineralization. *Advanced Materials*, 15(12), 959-970.

[14] Saldanha, R. B., Scheuermann Filho, H. C., Mallmann, J. E. C., Consoli, N. C., & Reddy, K. R. (2018). Physical–mineralogical–chemical characterization of carbide lime: An environment-friendly chemical additive for soil stabilization. *J. Mater. Civ. Eng*, 30(6), 06018004.

[15] Cardoso, F. A., Fernandes, H. C., Pileggi, R. G., Cincotto, M. A., & John, V. M. (2009). Carbide lime and industrial hydrated lime characterization. *Powder Technology*, 195(2), 143-149.

[16] Pelisser, G., Ferrazzo, S. T., Mota, J. D., Dos Santos, C. P., Pelisser, C., Rosa, F. D., & Korf, E. P. (2023). Rice husk ash-carbide lime as an alternative binder for waste foundry sand stabilization. *Environmental Science and Pollution Research*, 30(14), 42176-42191.

[17] Cardoso, F. A., Fernandes, H. C., Pileggi, R. G., Cincotto, M. A., & John, V. M. (2009). Carbide lime and industrial hydrated lime characterization. *Powder Technology*, 195(2), 143-149.

[18] Commey, A., & Mensah, M. (2019). An Experimental Study on the Use of Eggshell Powder as a pH Modifier: Production of Lime from Eggshells. *International Journal of Innovative Science and Research Technology*, 4(9).

[19] Onwubu, S. C., Mokgobole, M. U., Mdluli, P. S., & Mokhothu, T. H. (2022). Assessing the antibacterial properties of eggshell-titanium powder. *South African Dental Journal*, 77(8), 483-488.

[20] Busca, G., & Resini, C. (2006). Vibrational spectroscopy for the analysis of geological and inorganic materials. *Encyclopedia of Analytical Chemistry*: Applications, Theory and Instrumentation.

[21] Yanagisawa, Y., & Kashima, S. I. (2000). Interaction of CO with CaO surfaces: A TPD and FTIR study. *Surface science*, 454, 379-383.

[22] Blesa, M. J., Miranda, J. L., & Moliner, R. (2003). Micro-FTIR study of the blend of humates with calcium hydroxide used to prepare smokeless fuel briquettes. *Vibrational Spectroscopy*, 33(1-2), 31-35.

[23] Gomez-Vazquez, O. M., Correa-Piña, B. A., Zubieta-Otero, L. F., Castillo-Paz, A. M., Londoño-Restrepo, S. M., & Rodriguez-García, M. E. (2021). Synthesis and characterization of bioinspired nano-hydroxyapatite by wet chemical precipitation. *Ceramics International*, 47(23), 32775-32785.

[24] Cai, G. B., Chen, S. F., Liu, L., Jiang, J., Yao, H. B., Xu, A. W., & Yu, S. H. (2010). 1, 3-Diamino-2-hydroxypropane-N, N, N′, N′-tetraacetic acid stabilized amorphous calcium carbonate: Nucleation, transformation and crystal growth. *CrystEngComm*, 12(1), 234-241.

[25] Busca, G., & Resini, C. (2006). Vibrational spectroscopy for the analysis of geological and inorganic materials. *Encyclopedia of Analytical Chemistry*: Applications, Theory and Instrumentation.

[26] Tizo, M. S., Blanco, L. A. V., Cagas, A. C. Q., Cruz, B. R. B. D., Encoy, J. C., Gunting, J. V., ... & Mabayo, V. I. F. (2018). Efficiency of calcium carbonate from eggshells as an adsorbent for cadmium removal in aqueous solution. *Sustainable Environment Research*, 28(6), 326-332.

[27] Arami, M., Limaee, N. Y., & Mahmoodi, N. M. (2008). Evaluation of the adsorption kinetics and equilibrium for the potential removal of acid dyes using a biosorbent. *Chemical Engineering Journal*, 139(1), 2-10.

[28] Coates, J. (2000). Interpretation of infrared spectra, a practical approach. *Encyclopedia of analytical chemistry*, 12, 10815-10837.

[29] Ziembowicz, F. I., Bender, C. R., Frizzo, C. P., Martins, M. A. P., de Souza, T. D., Kloster, C. L., ... & Villetti, M. A. (2017). Thermodynamic insights into the binding of mono-and dicationic imidazolium surfactant ionic liquids with methylcellulose in the diluted regime. *The Journal of Physical Chemistry* B, 121(35), 8385-8398.

[30] Sachinath Mitra, fundamentals of optics, spectroscopic and X-ray mineralogy, Wiley Eastern Limited, New Delhi, 1989 pp.22

[31] Nagalakshmi, R., Kalpana, M., & Prabasheela, B. (2022, November). Eggshell derived calcium oxide–Synthesis and characterization. In *AIP Conference Proceedings* (Vol. 2446, No.1). AIP Publishing.

[32] He, M. (2005). *Slurry rheology of limestone and its effects on wet ultra-fine grinding* (Doctoral dissertation, Luleå tekniska universitet).

[33] Carr, R. L. (1976). Powder and granule properties and mechanics. M. Marchello, J. Gomezplata, A. (Eds.), Gas-solids Handling in the *Processing Industries*. Marcel dekker.

CHAPTER IV

APPLICATION

1. Introduction

An abrasive that is harder than the substrate is required to be able to change the substrate's surface structure. Furthermore, it is imperative that:

- Employ progressively finer grains to remove any imperfections or scuffs left by bigger grains (scratches are progressively smaller until they are invisible to the naked eye, giving the surface a polished appearance),

The goal of the polishing process is to achieve a surface finish comparable to enamel. To accomplish this, instruments equipped with abrasive particles ranging from 8 to 20 micrometers are employed. At the end of the process, surface irregularities are no longer noticeable under low magnification. It is crucial to clean the surface between each step, as residual particles from previous steps can introduce deeper irregularities [1].

The aim of this chapter is to evaluate the effectiveness of polishing agents formulated from two refined materials proposed in this study: eggshell powder and an industrial by-product. Furthermore, this chapter aims to investigate the feasibility of using an optimal mixture of these materials as a polishing agent.

These studies were conducted at a prosthetist facility in the town of Saida.

2. Procedure:

In this experimental procedure, the prostheses are initially washed with water to remove debris or contaminants and dried for 10 seconds with a clean cloth to ensure the removal of any surface contaminants and moisture. Following this, the initial weight of each prosthesis is measured and recorded with precision. Carefully, the surface is inspected for rough spots, flash, or other imperfections. The polishing agent slurry is prepared by mixing polishing agent powder with water in well-defined proportions. Subsequently, each prosthesis is subjected to a polishing process. The slurry is applied to a rag wheel mounted on a rotary lathe (1500rpm) wherein the polishing agent slurry is applied and the prostheses were polished for a duration of 1 minute. The prosthesis surface is polished in a circular motion, ensuring even coverage and focusing on areas with persistent roughness or scratches.

This polishing step is critical for assessing the impact of the polishing agent on the surface properties and overall weight of the prostheses. Finally, the prosthesis is rinsed thoroughly to remove powder residue then weigh

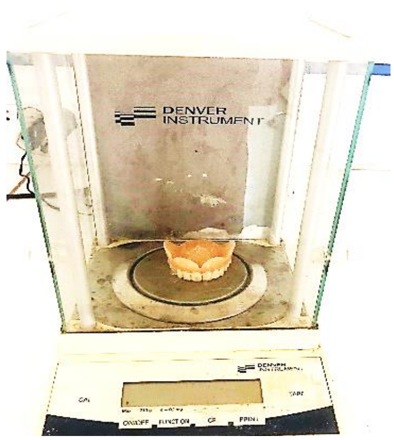
The surfaces of the experimental samples were observed by only one operator, who classified the surface states as*: matte, glossy, regular, and irregular*.

a) Washing

b) Polishing lathe TECHDENT

c) Polishing

d) Drying with a clean cloth after washing



e) Weighting

Figure IV-1:Polishing process

3. Development of the new abrasive materials

The prosthesis were polished using different particle sizes of the new abrasive materials (Table 01), in order to determine the particle size that produces the most effective surface finish.

According to the ASTM E11 standard for particle size classification **[2]**, two particle size distributions were adopted.

Table 01: Specification for particle size distribution ASTM.

Particle size category	**Particle size**
Fine	<=45µm
medium-fine to medium	45µm-0.3mm

Two new abrasive materials are introduced: eggshell powder and an industrial by-product. Each material was utilized with two specific particle size distributions, as detailed in the table 02.The 44µm particle size can be typified as medium dental abrasive material **[3].** Additionally, the potential of using a mixture of these two abrasive materials was investigated to assess their polishing effectiveness. The proportions used in these mixtures are provided in the table 02.

Table 02: Composition of the new abrasive materials used.

MATERIAL designation	**SIZE distribution**	**COMMERCIAL**	**EGGSHELL**	**BY PRODUCT**
MT0F	d <= 45µm	100	0	0
ME0F	d <= 45µm	/	100	0
MB0F	d <= 45µm	/	0	100
ME0MF	45µm<d<=0.3mm	/	100	/
MB0MF	45µm<d<=0.3mm	/	/	100
MEB50F	d <= 45µm	/	50	50
MEB50MF	45µm<d<=0.3mm	/	50	50
MEB20F	d <= 45µm	/	20	80
MEB20MF	45µm<d<=0.3mm	/	20	80
MEB80F	d <=45µm	/	80	20
MEB80MF	45µm<d<=0.3mm	/	80	20

Consistency of each slurry, prepared from the two particle size category, was achieved by mixing distillated water with new abrasive material in adequate proportions (Table 03)

Table 03: Material slurries

MATERIAL	**WATER (ml)**	**SAMPLE (g)**
MT0	5	3
ME0	5	4
MB0	5	5
MEB50	5	4.5
MEB20	5	4

The characteristics of the obtained slurries are presented in figure IV-2.

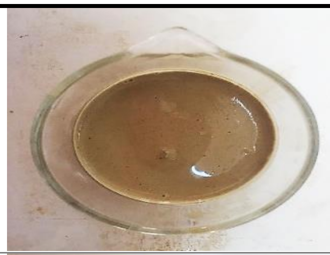
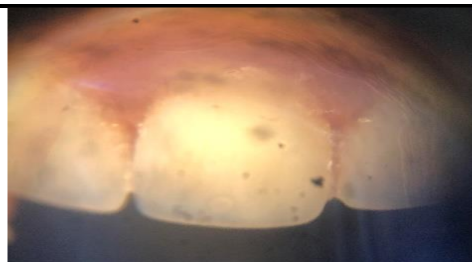
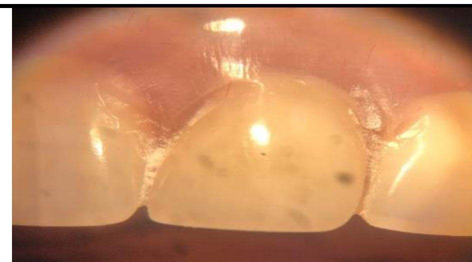
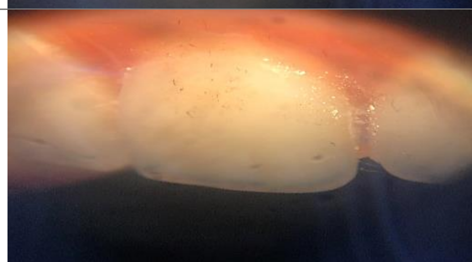
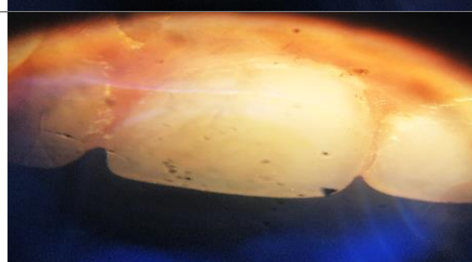
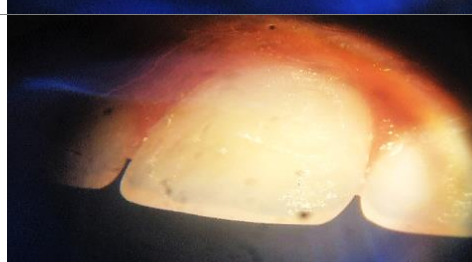
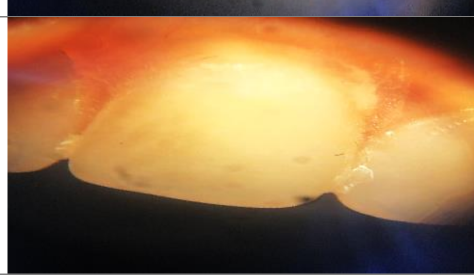
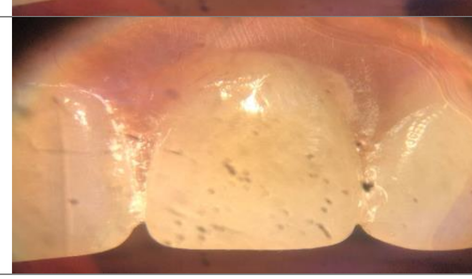
New abrasive material slurry	Visual aspect	Color	Odour	Texture
MT0		Beige	Odourless	rough texture Granular
ME0		Off-white	light odour	smooth texture Creamy, Light and Homogeneous
MB0		Gray	unpleased odour	Slightly rough texture Granular, Light
MEB50		Off-white	light odour	Slightly smooth texture Granular, Light and Creamy
MEB20		Gray	light odour	Slightly rough texture Pasty, Granular
MEB80		Light aray	light odour	Slightly rough texture Granular, Creamy

Figure IV-2: Visual Aspect and texture kind of the new abrasive material slurries

4. 1. Stereomicroscope analysis

After polishing the surface of the prosthesis, it was examined under a stereomicroscope to identify any irregularities. The results of this examination are presented in figure IV-3.

As illustrated in figure IV-3, stereo microscope images of the prosthesis specimens visibly confirmed the differences between the polished and unpolished surfaces.

Material	unpolished surface	polished surface
MT0		
ME0F		
MB0F		
ME0MF		
MB0MF		

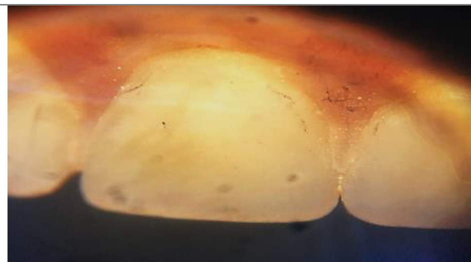
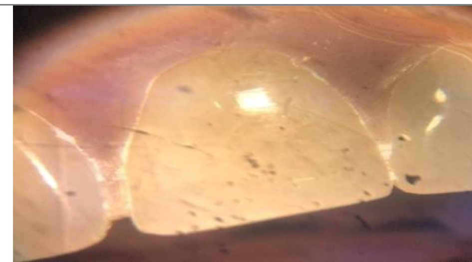
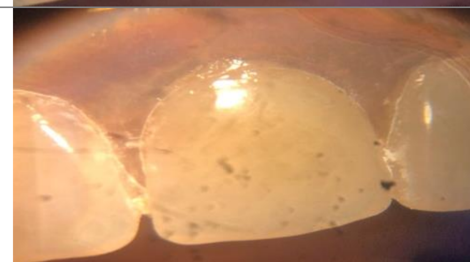
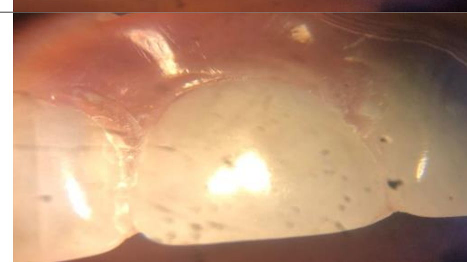
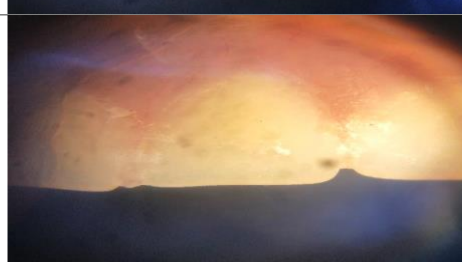
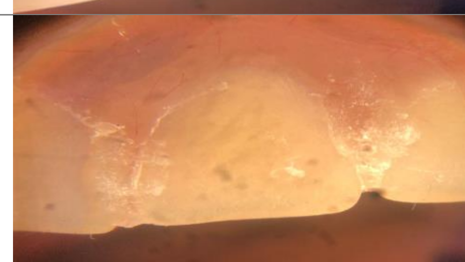
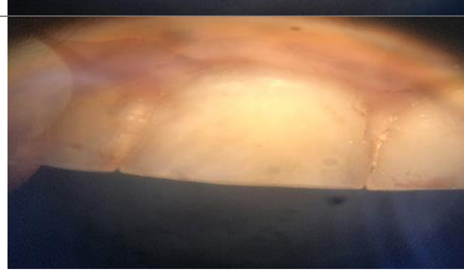
MEB50F		
MEB50MF		
MEB20F		
MEB20MF		
MEB80F		
MEB80MF		

Figure IV-3: Prosthesis before and after polishing process

The polished prosthesis surfaces by 80-20 Eggshell-Byproduct, despite the size grain distribution, showed slightly shiny surface.

Table 04: Prosthesis characteristics

Prosthesis	Surface Colour		Surface Roughness		Surface Brilliance	
	unpolished	polished	unpolished	polished	unpolished	polished
MT0	Slightly yellow	Off-white	Slightly rough	Slightly smooth	Dull	Slightly shiny
ME0F	Off-white	Off-white	Slightly smooth	smooth	Dull	brilliance
MB0F	Slightly yellow	Off-white	Slightly rough	slightly smooth	Dull	Shiny
ME0MF	Off-white	Off-white	rough	Slightly smooth	Dull	Dull
MB0MF	Off-white	Off-white	Slightly rough	Slightly smooth	Dull	Slightly shiny
MEB50F	Slightly yellow	Off-white	Slightly rough	Slightly smooth	Dull	brilliance
MEB50MF	Slightly yellow	Off-white	Slightly rough	Slightly smooth	Dull	Slightly shiny
MEB20F	Off-white	white	Slightly smooth	smooth	Dull	brilliance
MEB20MF	Off-white	white	Slightly smooth	smooth	Slightly shiny	brilliance
MEB80F	Slightly yellow	Off-white	Slightly rough	Slightly smooth	Dull	Slightly shiny
MEB80MF	Yellow with a little bit of black	yellow	rough	Slightly rough	Dull	Slightly shiny

In light of the aforementioned results, the new abrasive materials, significantly reduced the surface roughness of the polished area (figures IV-3 and 04), thereby confirming that the new polishing agents effectively reduce surface roughness.

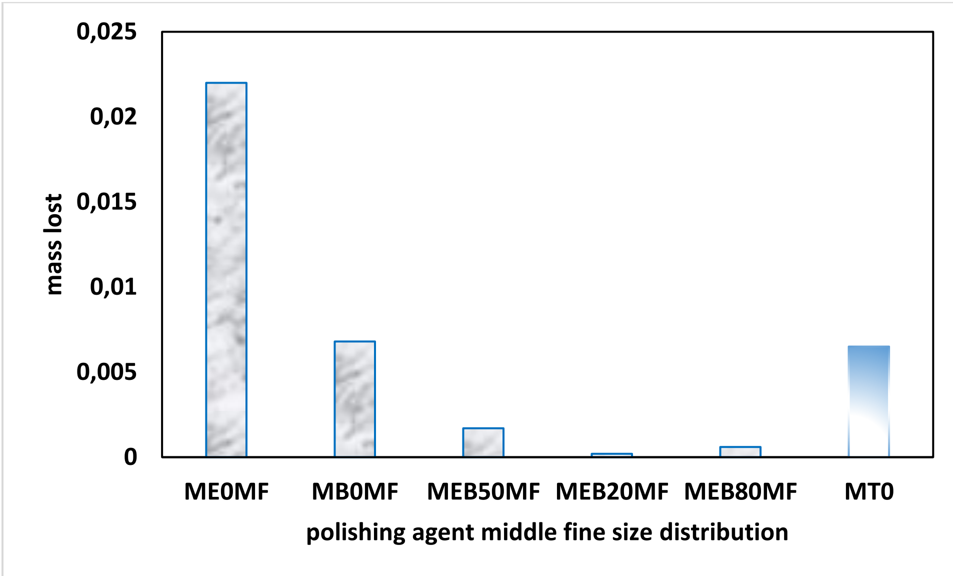


Figure IV-4: Lost mass Evolution of polished prosthesis by middle fine size abrasive materials

According to the lost mass obtained from polishing action (table 05) showed a significant difference between new abrasive materials and commercial product; this significant difference may be due to variation in their composition (mineral content) [4] that increase the abrasive resistance of the eggshell powder and industrial byproduct.

This may be due also to the particle size of the material that when the polisher material has smaller particle which mean increase in wear which lead to increase in polishing mechanism [5]. Figures IV-4 and IV-5 disclose that fine size distribution of both abrasive materials eggshell and by-product due to the abrasion power of these materials which may abrade easily compared to commercial product.

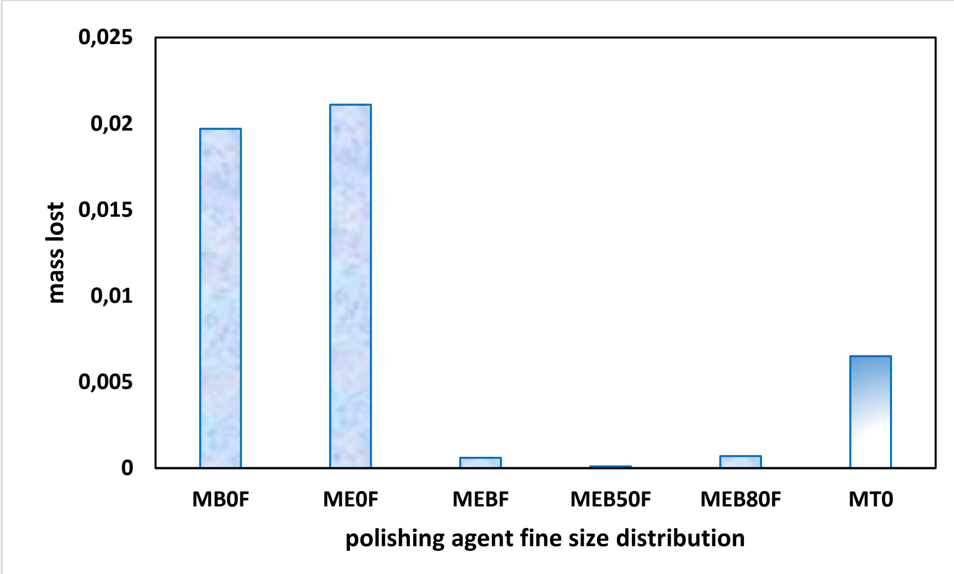


Figure IV-5: Lost mass Evolution of polished prosthesis by fine size abrasive materials

Importantly, the Δm values obtained after polishing by the new abrasive material mixtures were below the value resulted by commercial product (0.0065g) (table 05). As commercial product, the results of this study revealed that the new abrasive material composed by fifty-fifty eggshell - by-product is likely to produce polished surfaces.

Table 05: lost mass of prosthesis after polishing process

new abrasive material	lost mass of specimen		
	unpolished specimen mass m0	polished specimen mass m1	lost mass Δm m0-m1
MT0	17.1489	17.1424	0.0065
ME0F	12.9963	12.9752	0.0211
MB0F	11.7762	11.7565	0.0197
ME0MF	14.4184	14.3964	0.022
MB0MF	15.0467	15.0399	0.0068
MEB50F	17.0710	17.0709	0.0001
MEB50MF	13.7591	13.7574	0.0017
MEB20F	15.6791	15.6785	0.0006
MEB20MF	12.2748	12.2746	0.0002
MEB80F	10.9829	10.9822	0.0007
MEB80MF	14.1737	14.1731	0.0006

Moreover, the fine particle sizes of the new polishing agents contributed to the significant differences in the mass loss (figure IV-5). Abrasive materials of smaller particle sizes, as present in eggshell and by-product with size distribution <=45µm (Fine size grain), quickly expose newly-formed and sharper particles during the abrasion process and subsequently reduce surface roughness rapidly [6]. This conforms to the principles of abrasion that is harder materials come into frictional contact with surface: the prosthesis in this study [7].

All the polishing materials included eggshell powder (figure IV-6) proffered brilliance to the polished surface with Fine size distribution grain, expected when 20% of eggshell are substituted by byproduct.

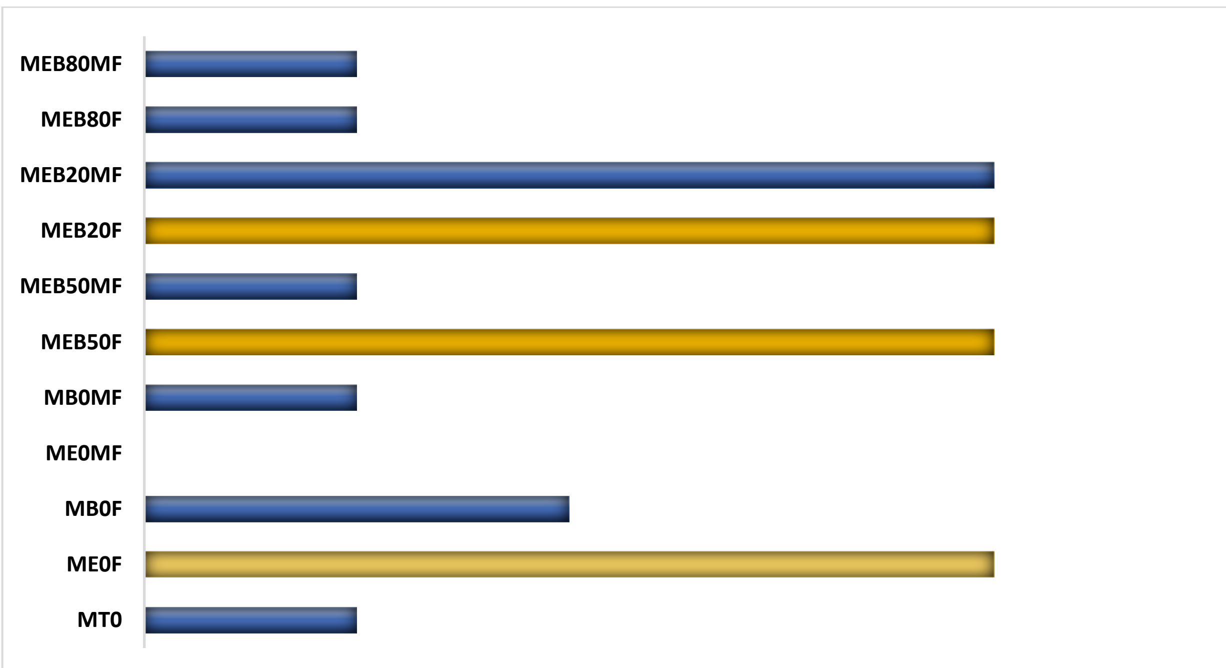


Figure IV-6: Brilliance level of the polished surface prosthesis

The results are better than the commercial product. These new abrasive materials whatever the size grain distribution showed brilliance when there are composed by 20-80 (eggshell-byproduct). Although, Eggshell powder with middle fine size distribution grain, as polishing agent didn't ameliorate the brilliance of the specimen whereas with the fine one, the brilliance is guaranteed.

The results of this study (figure IV-7) confirm that polishing process with the new abrasive materials can reduce surface roughness. In clinical practice highly smoothened surfaces are generally good indicators of surface luster of polished dental prostheses [8].

Overall, the results recorded in figure IV-7 show that surface roughness, polished with fine eggshell powder is considerably lower than those observed in the commercial product-polished specimens. Despite this, byproduct-polished specimen show that is more likely to produce a more highly polished dental prosthesis when 20% eggshell is added. So, the inclusion of eggshell powder in byproduct abrasive material could have contributed to ameliorate the surface smoothing.

Figure IV-7: Roughness level of the polished surface prosthesis

Ordu and al. [9] reveal that eggshell power had smaller particle sizes when compared to pumice, the most used as polishing agent in dental domain. It is reported that abrasive materials of smaller particle sizes created newly formed and sharper particles faster during the abrasion process, which in turn reduces the surface roughness of dental prostheses [10].

Arguably, the improved white color surfaces of the prosthesis specimens could be attributed by the mixture eggshell-byproduct composition of the new polishing agent (figure IV-8). Contrary to eggshell powder, byproduct gives a white color to the polished prosthesis regardless its particle size distribution if it introduces with a portion of 80%in the mixture eggshell-byproduct.

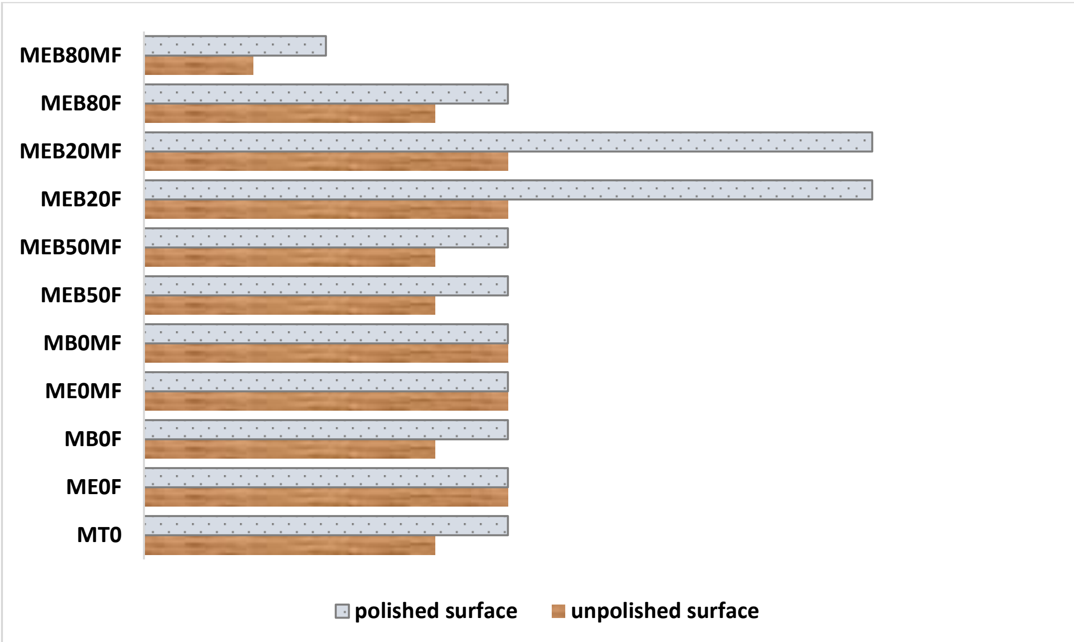


Figure IV-8: Evolution of White colour level of the polished surface prosthesis

5. Conclusion

Overall, the salient features of this study demonstrated that the new abrasive materials effectively reduced the surface roughness of the prosthesis specimens to below the prosthesis polished by commercial product action.

This further confirmed that the fine particle size of the new abrasive materials used in this study provided better polished surfaces.

Investment materials act as a good polishing material prosthesis material rather commercial product.

The mixture 20-80% (eggshell powder-byproduct decrease the surface roughness. The white color is accentuated by the same mixture as the brilliance of the specimen surface.

References

[1] Maalhagh-Fard, A., Wagner, W. C., Pink, F. E., & Neme, A. M. (2003). Evaluation of surface finish and polish of eight provisional restorative materials using acrylic bur and abrasive disk with and without pumice. *Operative dentistry*, *28*(6), 734-739.

[2] Roostaei, M., Soroush, M., Hosseini, S. A., Velayati, A., Alkouh, A., Mahmoudi, M., Ghalambor, A. & Fattahpour, V. (2020, February). Comparison of various particle size distribution measurement methods: role of particle shape descriptors. In *SPE International Conference and Exhibition on Formation Damage Control* (p. D022S011R004). SPE.

[3] Anusavice, K. J., Shen, C., & Rawls, H. R. (Eds.). (2012). *Phillips' science of dental materials*. Elsevier Health Sciences.

[4] Sharmista, S., Gudala, S. K., Yalavarthy, R., Srinivas, K., & Krishna, H. (2020). Surface Roughness of Acrylic Denture Base Resins Polished with Different Abrasive Agent: An In vitro Study. *International Journal of Dental Materials*, 2(3), 91-97.

[5] M. Aldwimi, I., Md. Akil, H., Abdul Hamid, Z. A., & Alhareb, A. O. (2022, October). Evaluation of Flexural Strength Properties and Surface Roughness of Poly (Methyl Methacrylate) Denture Base Material Reinforced with Different Nanotubes' Fillers. In *Asian Workshop on Polymer Processing* (pp. 165-182). Singapore: Springer Nature Singapore.

[6] Hanna, B. A., Abd Al-Majeed, A. E., & AbdulRazaak, W. (2008). Effect of different dental materials on the surface roughness of acrylic resin (A comparative in vitro study). *MDJ*, *5*, 281-285.

[7] Ordu, J. I., George, T., & Okafo, S. E. (2023). Formulation and evaluation of oral paste made using calcium carbonate extract from the shell of Busicon carica (Buccinidae). IOSR Journal of Pharmacy and Biological Sciences, 18(6), 14-23.

[8] Kassoob, A. H., & Yaseen, I. N. (2022). The effects of polishing techniques on surface roughness of de-flex denture base. *Journal of Techniques*, 4(3), 65-69.

[9] Gungor, H., Gundogdu, M., & Duymus, Z. Y. (2014). Investigation of the effect of different polishing techniques on the surface roughness of denture base and repair materials. *The Journal of prosthetic dentistry*, 112(5), 1271-1277.

[10] Eirich, F. R. (1976). The role of friction and abrasion in the drilling of teeth. In *The Cutting Edge: Interfacial Dynamics of Cutting and Grinding: Proceedings of a Symposium Sponsored by the American Association for the Advancement of Science and Supported in Part by National Institute of Dental Research and National Science Foundation*. US Department of Health, Education, and Welfare, Public Health Service, National Institutes of Health, National Institute of Dental Research.

GENERAL CONCLUSION

General conclusion:

The purpose of polishing dental prostheses is to create smoother tooth surfaces, enhancing their appearance, reducing adhesion, and preventing deterioration. This process progressively eliminates scratches using finer abrasives like dental pumice, rotating wheels, and polishing compounds to achieve a glossy finish. Dental pumice, a powdered abrasive, is a crucial material widely utilized in dentistry for polishing dental prostheses.

Our study aims to create a polishing agent for dental prostheses with superior quality compared to those currently available in the market. According to the market product composition, the product's CaO rate is less than 10%, indicating that it cannot be identified as pumice. The percentage of humidity indicated that this substance is hydrophilic. The product has a very poor flowability character. Less than 80 µm makes up its granulometry. The pH of the final slurry is 9.1.

We conducted a characterization of the materials that will be part of our new polishing agent. Based on the following findings, the examination of industrial waste suggests using it as a polishing agent. Mineral elements such as calcite and portlandite are present in significant amounts in carbide lime. The suspension of the pH by-product is 12. Excellent cleaning qualities are offered by an abrasive precipitated calcium carbonate that isn't too abrasive or harmful to tooth surfaces or gums. The predominant particle size of the precipitated carbide lime is around 80 µm. The pH of the calcium hydroxide slurry remains constant.

One of the main ingredients for our polishing agent will be the valorization of eggshells, which counts as an innovative idea, supported by the following results:

An examination of eggshell powder showed that it contains a lot of carbonate. By its very nature, calcium carbonate, the main inorganic mineral, is a poor source of nutrition for microorganisms to colonize. The eggshell's mean average size was measured between 0.02 and 0.2 µm. Particles of eggshell are grouped together. The fineness of the powder particle size is shown by the granulometry examination. The powder is cohesive and has poor coulability.

In other words, Part One's main goal was to determine whether eggshells and industrial byproducts might be used as an alternative abrasive material by analyzing them. The materials' composition of distinct water-absorbing calcite was confirmed by both FTIR and XRD studies. These findings validated the possibility of bacterial-free materials. The improved physical and chemical properties of the samples validate their use as a substitute for pumice in the abrasive material used to polish removable dental equipment in terms of their granulometry and mineral makeup.

By showcasing the abilities of the suggested materials to lessen the surface roughness of the prosthesis area, the following test section of our study verifies the research concept.

Moving on to the application results, we found that in general, this study's key findings showed that the novel abrasive materials successfully lowered the surface roughness of the prosthesis specimens to a level lower than those polished by purchased commercial product. This further demonstrated that the novel abrasive materials utilized in this investigation produced better polished surfaces due to their finer particle size. These invested materials are more effective as prosthetic materials for polishing than commercial products. The combination of 20–80% (a byproduct of eggshell powder) reduces surface roughness, highlighting the specimen surface's brightness and white color.

In the end, this modest effort demonstrates that we could producte a low cost new abrasive material as polishing agent in dental domain. Our product could surpassing the imported one, thereby bringing us one step closer to realizing our idea and achieving our goals "***creating our startup***".