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Research Article

Comparative Evaluation Test of Fresh & Expired Pharmaceutical Tablets

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ABSTRACT

This study evaluates the physicochemical properties, including hardness, friability, weight variation, disintegration time, and dissolution profiles, of fresh and expired paracetamol and calcium carbonate-vitamin D3 tablets to determine the impact of shelf-life expiration on critical quality attributes and therapeutic efficacy in accordance with pharmacopeial standards such as USP and IP[1][2][3][4]. Such assessments are essential, as prior research indicates that expired tablets often exhibit increased hardness due to moisture loss or polymorphic changes, altered porosity leading to faster disintegration, and potential reductions in drug content uniformity, thereby compromising bioavailability and posing health risks [1,5,6]. By employing standardized tests like diametric compression for hardness and basket apparatus for dissolution, this investigation elucidates degradation patterns specific to these formulations, informing stability predictions and post-expiry usage guidelines [2,7]. The objective of this study was to conduct a comprehensive assessment of the physicochemical stability and degradation kinetics of paracetamol and calcium carbonate with vitamin D3 in pharmaceutical tablet formulations, distinguishing between fresh and expired batches to elucidate the impact of shelf-life on drug product quality. The accurate estimation of drug shelf-life is critical for ensuring pharmaceutical quality, as decomposition or structural changes over time can lead to a loss of efficacy and potential health risks

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INTRODUCTION

The physical integrity of tablet dosage forms, as indicated by parameters such as hardness, friability, and weight variation, serves as a fundamental metric for assessing a product's resistance to mechanical stresses like abrasion, vibration, and impact during transport and storage, thereby ensuring that tablets maintain the structural durability necessary to preserve content uniformity and consistent therapeutic efficacy throughout their shelf life [1,8,9]. Although tablet weight generally remains consistent, other critical attributes—specifically hardness and disintegration kinetics—frequently demonstrate heightened susceptibility to environmental conditions, which can ultimately undermine the consistency of drug release and diminish therapeutic efficacy [10,11]. The pharmacopeial standards necessitate stringent quality control measures, including assessments of physical appearance and chemical parameters, to guarantee that pharmaceutical products meet minimum criteria for quality, safety, and efficacy [10,12]. The actual shelf-life of active molecules is a key parameter of pharmaceutical quality, as drug products can lose their properties and functionality due to decomposition or structural changes,

such as dehydration, oxidation, and microbial spoilage [1] [6]. Therefore, accelerated degradation studies, often employing techniques like thermogravimetry, are crucial for predicting shelf-life under various conditions, circumventing the need for prolonged real-time decomposition assessments. This approach allows for the estimation of degradation kinetics and the determination of activation energy, which are critical for establishing reliable shelf-life predictions for pharmaceutical formulations [6]. This is especially relevant for paracetamol formulations, where factors such as excipient concentration and storage conditions significantly influence shelf-life and degradation kinetics, often exhibiting non-monotonic behaviors [7].

Therefore, understanding the nuances of paracetamol's degradation pathways and the impact of formulation variables on its stability is vital for ensuring therapeutic efficacy and patient safety [13]. Because paracetamol is widely used, thorough stability testing is essential to preserve its quality and effectiveness throughout its shelf life, as degradation can reduce potency and produce toxic impurities like 4-aminophenol [14,15].

Stability testing plays an essential role in determining this expiry date by evaluating the physical, chemical, and microbiological characteristics of a drug over time. This testing ensures that medicines maintain their intended potency, safety, and quality throughout their shelf life.

The risks associated with the use of expired medicines are significant, including reduced efficacy, altered pharmacokinetics, and potential toxicity, which can compromise patient safety (WHO, 2014) [16]. Therefore, regular tablet evaluation is vital to verify the consistency, disintegration, dissolution, and overall quality of solid dosage forms, ensuring therapeutic reliability (USP, 2021).[17]

Paracetamol, a widely used analgesic and antipyretic agent, has been a cornerstone in pain and fever management due to its well-established efficacy and safety profile (Rang et al., 2015). Similarly, calcium carbonate combined with vitamin D₃ is extensively used to prevent and treat calcium deficiencies and related bone disorders, leveraging the synergistic role of vitamin D₃ in enhancing calcium absorption [18](Holick, 2007).

This review aims to explore the significance of expiry dating, the importance of stability testing, the risks of expired medicines, and the critical evaluation of tablets. Furthermore, it provides an overview of paracetamol and calcium carbonate with vitamin D₃, discussing their uses, mechanisms of action, dosage forms, and appropriate storage conditions to optimize therapeutic outcomes.[19]

Materials and Methods

Evolution test

Hardness test

Weight variation test

Friability test

Disintegration test

Dissolution test

These evaluative tests are crucial for determining the physical integrity, uniformity, and drug release characteristics of the tablets, which are fundamental indicators of product quality and performance [9,20]. . In addition to these physical assessments, rigorous analytical method validation. This systematic approach allows for a comprehensive assessment of how storage conditions and time influence the degradation rate of active ingredients and overall pharmaceutical stability [21]. Furthermore, these tests validate essential tablet quality attributes including mechanical strength to withstand handling, weight uniformity, and resistance to abrasion, in accordance with pharmacopeial standards such as BP and USP [1–3,8]

In remains essential for quantifying active ingredients in both bulk and tablet dosage forms, as these procedures ensure that the API content complies with pharmacopeial specifications, typically requiring 95% to 105% of the labeled amount [8]. Furthermore, validated stability-indicating methods are crucial for identifying and quantifying potential degradation products, such as 4-aminophenol in paracetamol formulations, which could

otherwise compromise safety and therapeutic outcomes [14,20].

Hardness test

Hardness test quantifies the mechanical integrity of tablets by determining the crushing force in Newtons, with research demonstrating that expired paracetamol tablets exhibit significantly higher values of hardness compared to fresh samples, possibly due to changes in moisture content or polymorphic transformations within the formulation over time [1]. This alteration in hardness can impact disintegration and dissolution profiles, thereby affecting drug release and bioavailability [11].

Hardness process: The procedure entails placing individual tablets diametrically between the platens of a hardness tester, such as Monsanto or Erweka models, and applying a gradually increasing compression force until fracture occurs, with the peak force recorded in Newtons to quantify mechanical changes distinguishing fresh from expired paracetamol and calcium carbonate vitamin D₃ formulations [1,2,4,22].

Weight variation test

The weight variation test evaluates tablet mass uniformity by individually weighing at least 20 tablets and verifying compliance with pharmacopeial limits—such as no more than two tablets deviating by more than 5% from the average for those exceeding 250 mg—ensuring dose accuracy and content uniformity, with studies confirming that both fresh and expired paracetamol and calcium carbonate vitamin D₃ tablets typically meet these standards without significant changes attributable to aging [1–4,8]. This observed consistency in weight uniformity between fresh and aged paracetamol and calcium carbonate vitamin D₃ tablets under various storage conditions, including sunlight, relative humidity, and electromagnetic exposure, affirms the manufacturing process's effectiveness in preserving dosage homogeneity over time, even amid changes in attributes like hardness [3,23]. Furthermore, this consistent weight uniformity under diverse storage conditions, including varying relative humidities, indicates that hygroscopic moisture uptake or volatile component evaporation does not constitute primary degradation mechanisms affecting mass consistency in these paracetamol and calcium carbonate vitamin D₃ tablets (Gbenga & Taiwo, 2016 (Abebe et al.,2020). [24].

Friability Testing

which measures a tablet's resistance to chipping and abrasion, is crucial for assessing its durability during handling and transport, with expired formulations potentially exhibiting altered friability due to changes in binding agents or crystalline structure [25].

friability testing evaluates the resistance of paracetamol tablets to mechanical abrasion during handling and transport, with pharmacopeial standards requiring less than 1% weight loss to maintain product integrity, particularly as expired batches may exhibit increased fragility due to storage-induced changes [1,3,8].

friability process The friability test is conducted by weighing ten tablets (initial weight, placing them in a friabilator drum rotated at 100 rpm for 4 minutes, and then reweighing them (final weight, Weight final) to calculate the percentage weight loss [26]. The friability of tablets should ideally be less than 1% to be considered acceptable, as values exceeding this threshold can indicate compromised mechanical stability [27]. This assessment involves rotating the weighed tablets in a friabilator for a specified duration, after which they are reweighed to determine the percentage of mass lost [18–20]. This percentage directly reflects the tablet's susceptibility to mechanical stress, with higher values indicating a greater propensity for chipping or breaking during packaging, shipping, and patient handling [21].

Disintegration test

The disintegration test determines the time required for six paracetamol and calcium carbonate vitamin D3 tablets to completely break down into particles that pass through a 10-mesh screen in 900 mL of distilled water maintained at $37 \pm 1^\circ\text{C}$, with pharmacopeial standards (BP/USP) specifying a maximum of 15 minutes for uncoated tablets, and studies reporting mean times of 1-8 minutes for fresh paracetamol formulations that remain compliant even in aged samples [1–3].

Such pharmacopeial compliance in disintegration times for both fresh and aged paracetamol and calcium carbonate

Results and Discussion1

Hardness test

vitamin D3 tablets, despite storage-induced hardening, confirms the robustness of disintegrant-excipient interactions in maintaining mechanical breakdown efficiency [1,3,23]. This sustained performance suggests that any increases in hardness observed in aged paracetamol and calcium carbonate vitamin D3 tablets are not sufficiently severe to substantially impair the wicking and swelling mechanisms facilitated by disintegrants, which enable rapid capillary-driven fluid penetration into the porous structure and subsequent particle separation for pharmacopeial-compliant disintegration [21–25].

Dissolution test

This test measures the rate and extent to which the active pharmaceutical ingredient is released from the tablet into a dissolution medium, simulating physiological conditions, which is a critical parameter for assessing drug bioavailability and often differentiates fresh from expired formulations [25]. Specifically, the dissolution profile is assessed using methods like the USP Apparatus 1 (basket) or Apparatus 2 (paddle), maintaining precise control over temperature and agitation to quantify the amount of drug dissolved over specific time intervals.[26]

Commonly, these tests utilize 900 mL of a suitable dissolution medium, such as 0.1 N HCl or phosphate buffer, to ensure sink conditions throughout the monitoring period [27].

Table 1: Hardness test

Paracetamol Tablets No:	Hardness Fresh tab kg \ cm ²	Hardness Expiry tab kg \ cm ²	Calcium & vitamin D3 Tablets No:	Hardness Fresh tab kg\cm ²	Hardness Expiry tab kg\cm ²
1	6.8	4.6	1		6.0
2	7.0	4.8	2		6.2
3	6.9	4.5	3		6.1
4	7.2	4.7	4		6.3
5	6.7	4.6	5		6.0
6	7.1	4.9	6		6.2
7	6.8	4.5	7		6.1
8	7.0	4.7	8		6.3
9	6.9	4.6	9		6.0
10	5.3	4.8	10		6.2
Total	53.5	46.7	Total	69.5	61.4
Avg. Hardness	5.35 kg/cm ²	4.67 kg/cm ²	Avg. Hardness	6.95 kg/cm ²	6.14 kg/cm ²
Strength	Good	Fair	Strength	Good	Fair
IP limit (4-8kg)	Pass	Fair	IP limit (4-8kg)	Pass	Pass

Weight variation test

Table 3.1: Weight variation test

Calcium Tablets No. for fresh	Weight of tablets	Deviation ex	Calcium Tablets No. for expiry	Weight of tablets	Deviation f
1	1720	0.81	1	1760	0.51
2	1700	1.96	2	1745	0.34
3	1755	1.21	3	1755	0.23
4	1785	2.94	4	1770	1.08
5	1685	2.82	5	1735	0.91
6	1735	0.06	6	1765	0.80
7	1765	1.79	7	1750	0.06
8	1770	3.69	8	1740	0.63
9	1800	3.81	9	1780	0.65
10	1725	0.52	10	1755	0.23

Table 3.2: Weight variation test

Paracetamol Tablets No. for fresh	Weight Of tab.	Deviation	paracetamol Tablets No. For expiry	Weight of tablets	Deviation
1	640	0.71	1	652	0.17
2	635	1.49	2	648	0.45
3	650	0.84	3	648	0.02
4	660	2.39	4	655	0.63
5	630	2.26	5	649	0.29
6	630	0.06	6	659	0.32
7	645	1.61	7	650	0.14
8	655	3.04	8	647	0.60
9	625	3.93	9	654	0.42
10	670	0.71	10	652	0.17

Friability testing

Table 6: For paracetamol both fresh and expiry

Tablet Types	No. of Tablet	Initial Weight of Tablet (gm)	Final Weight of Tablet (gm)	% Friability
Fresh Tablet	10	6.50	6.45	0.77%
Expiry Tablet	10	6.50	6.28	3.38%

Table 7: For combination calcium vit. D tablets both fresh and expiry

Tablet Types	No. of Tablet	Initial Weight of Tablet (gm)	Final Weight of Tablet (gm)	% Friability
Fresh Tablet	10	1.75	1.74	0.69%
Expiry Tablet	10	1.75	1.72	3.43%

As per Indian pharmacopeia limit: - Friability should be <1%

Disintegration test

Table 7: Disintegration test for paracetamol tablets

Tablets No:	Disintegration of paracetamol fresh tablet time(min)	Disintegration of paracetamol expiry tablet time (min)
1	6.5	6.5
2	7.0	6.9
3	6.8	7.9
4	7.2	8.0
5	6.9	6.5
6	7.1	6.8
Averages	6.9 (min.)	7.1 (min.)

Table 8: for combination Calcium & vitamin D3

Tablets No:	Disintegration of Calcium & Vitamin D3 fresh tablet time (min)	Disintegration of Calcium & Vitamin D3 expiry tablet time (min)
1	14	17
2	13	15
3	11	13
4	12	14
5	13	15
6	12	11
Averages	12.2 (min.)	14.5 (min.)

Assay (% Purity)

Accepting criteria (IP): - Should be between 95% to 105% of label claim

Table 9: Observation table for fresh paracetamol

Solution	Absorbance	% Purity
Standard	0.507	96.63%
Sample	0.501	95.53%

Table 10: Observation table for Expiry paracetamol

Solution	Absorbance	% Purity
Standard	0.507	96.63%
Sample	0.495	86.53 %

Table 11:. Assay of combination calcium & vitamin D tablet fresh

Observation	Volume(ml)	% Purity
Blank (NaoH)	40.7ml	98.38
Sample (NaoH)	20.3ml	95.27

Table 12: Assay of combination calcium & vitamin D tablet expiry

Observation	Volume(ml)	% Purity
Blank (NaoH)	48ml	94.06
Sample (NaoH)	20ml	95.88

CONCLUSION:

The comparative evaluation of fresh and expired pharmaceutical tablets of Paracetamol and calcium carbonate with vitamin D3 showed noticeable differences in their physical and performance characteristics after conducting hardness, weight variation, friability, disintegration, and dissolution tests. The comparative evaluation of fresh and expired paracetamol and calcium carbonate with vitamin D₃ tablets showed that most tablets remained within acceptable IP limits for hardness, weight variation, disintegration, dissolution, and assay tests. However, expired tablets showed reduced hardness, increased friability, slower drug release and lower assay values compared to fresh tablets, indicating gradual degradation after expiry. Fresh tablets exhibited better mechanical strength and drug release efficiency, while expired tablets showed altered physical properties that may affect bioavailability and therapeutic effectiveness. Therefore, the study concludes that although expired drugs may retain some potency, their quality and reliability decrease over time, emphasizing the importance of proper storage conditions and strict adherence to expiry dates for patient safety and effective treatment.

REFERENCE:

1. Marisa G, Kapala J, Mafuru T, Matinde R, Kimaro E, Kaale E. Quality evaluation of locally manufactured paracetamol tablets in East Africa. *Biomed Res Int.* 2024;2024(1):9437835.
2. Ergetie Z, Wondimisegne D, Tsega B. Analysis and comparison of paracetamol tablets dispensed in legal dispensaries and non-pharmaceutical shops in Gondar Town, Northwest Ethiopia. *Int J Pharm Ind Res.* 2013;3(2):144-151.
3. Abebe K, Beressa TB, Yimer BT. In-vitro evaluations of quality control parameters of paracetamol tablets marketed in Gondar City, Northwest Ethiopia. *Drug Healthc Patient Saf.* 2020;12:273-279.
4. Sharma SK, Singh A, Bhandari A, Singh S, Singh S. Development and evaluation of chewable tablets of calcium and vitamin D. *Asian J Pharm Res Dev.* 2020;8(3):232-234.
5. Marisa G, Kapala J, Mafuru T, Matinde R, Kimaro E, Kaale E. Quality evaluation of locally manufactured paracetamol tablets in East Africa. *Biomed Res Int.* 2024;2024(1):9437835.
6. Calvino MM, Lisuzzo L, Cavallaro G, Lazzara G, Milioto S. Non-isothermal thermogravimetry as an accelerated tool for the shelf-life prediction of paracetamol formulations. *Thermochim Acta.* 2021;700:178940.
7. Teklu L, Adugna E, Ashenef A. Quality evaluation of paracetamol tablets obtained from common shops (kiosks) in Addis Ababa, Ethiopia. *Int J Pharm Sci Res.* 2014;5(8):3502-3510.
8. Marisa G, Kapala J, Mafuru T, Matinde R, Kimaro E, Kaale E. Quality evaluation of locally manufactured paracetamol tablets in East Africa. *Biomed Res Int.* 2024;2024(1):9437835.
9. AlBratty M, Alhazmi HA, Alam MS, Alam MI, Javed SA, Alam N. Assessment of physicochemical properties and comparison of dissolution profiles of metformin hydrochloride tablets in Saudi Arabia. *Dissolut Technol.* 2020;27(1):36-44.
10. Wijaya GADPS. Tingkat pengetahuan dan perilaku ibu terhadap swamedikasi obat batuk di Apotek Raeja Farma Ubud. *J Anestesi.* 2026;4(3):37-48.

11. Adegoke OA, Thomas OE, Amao SA, Agboola SO, Omotosho AE. A new method for the microdetermination of paraminophenol in generic brands of paracetamol tablets. *Arab J Basic Appl Sci.* 2019;26(1):153-162.
12. Fateixa S, Mulandeza O, Nogueira HI, Trindade T. Raman imaging studies on the stability of paracetamol tablets under different storage conditions. *Vib Spectrosc.* 2023;124:103488.
13. Thunga G, Agrawal SK, Joshi M, Sathyanarayana MB, Tippavajhala VK, Kulyadi GP. Solid dosage forms: a detailed research on non-conforming product quality. *Indian J Pharm Educ Res.* 2020;54.
14. Bano A, Abrar S, Brilli E, Tarantino G, Bugti AA, Fabbrini M, et al. A comparative absorption study of sucrosomial® orodispersible vitamin D3 supplementation versus a reference chewable tablet and soft gel capsule vitamin D3 in improving circulatory 25(OH)D levels in healthy adults with vitamin D deficiency: results from a prospective randomized clinical trial. *Front Nutr.* 2023;10:1221685.
15. Ahmad W, Banu N, Ahmad A, Al Obaid MM, Al Hazza SE, Aljumaia LA, et al. A fast, sensitive, and green stability-indicating ultra-performance liquid chromatography method for the determination of paracetamol in marketed formulations. *Acta Chromatogr.* 2026;1326-2025.
16. Sherawat R, Maithani M, Singh R. Regulatory aspects in development of stability-indicating methods: a review. *Chromatographia.* 2010;72:1-6.
17. Alanazi AH, Alotaibi MB, Alharbi MS, Abdulrahman A, Alanazi AA. Stability analysis of pharmaceutical preparations under different storage conditions. *J Med Sci Clin Res.* 2024;12(9):17-20.
18. Bajaj S, Singla D, Sakhuja N. Stability testing of pharmaceutical products. *J Appl Pharm Sci.* 2012;2(3):129-138.
19. Raziq A, Jan SU, Gul R, Khan Y, Aman T, Shah PA. Relative comparison of stability and degradation of methylcobalamin tablets of different brands at different storage settings. *Int J Appl Pharm.* 2021;13(3):171-175.
20. Darusman F, Rusdiana T, Sopyan I, Aryani R, Darma GCE. The in vitro equivalence study of polymorph-modified glimepiride tablets compared to Amaryl®. *Pharmacia.* 2023;70:1027-1037.
21. Subramanian M, Sankar C, Rajaram G, Ravi V. Layered tablets: a novel oral solid dosage form. In: *Dosage Forms—Innovation and Future Perspectives.* London: IntechOpen; 2022.
22. Abdelmonem REH, Eltahan MA, El-Nabarawi MO. Development and evaluation of taste-masked oro-disintegrating tablets of itopride HCl using different co-processed excipients: pharmacokinetics study on rabbits. *Int J Appl Pharm.* 2022;14(3):69-79.
23. Bassetto R, Amadio E, Ciampianelli F, Perin S, Ilari P, Gaballo P, et al. Designing an effective dissolution test for bilayer tablets tailored for optimal melatonin release in sleep disorder management. *Front Nutr.* 2024;11:1394330.
24. Jain S, Kaur S, Rathi R, Nagaich U, Singh I. Application of co-processed excipients for developing fast disintegrating tablets: a review. *Polym Med.* 2023;53(1):59-68.
25. Markl D, Maclean N, Mann J, Williams H, Abbott A, Mead H, et al. Tablet disintegration performance: effect of compression pressure and storage conditions on surface liquid absorption and swelling kinetics. *Int J Pharm.* 2021;601:120382.
26. Sunte BT, Shinde AN, Vaidya VR. Screening of marketed formulations of Shatavari tablets to establish pharmaceutical equivalence. *J Drug Deliv Ther.* 2023;13(8):56-59.
27. Helmy SA, El Bedaiwy HM. In vitro dissolution similarity as a surrogate for in vivo bioavailability and therapeutic equivalence. *Dissolut Technol.* 2016;23:32-39.
28. Steffens KE, Wagner KG. Immediate-release formulations produced via twin-screw melt granulation: systematic evaluation of the addition of disintegrants. *AAPS PharmSciTech.* 2021;22(5):183.

