Int. j. adv. multidisc. res. stud. 2026; 6(5):907-911

Received: 06-08-2026
Accepted: 16-09-2026

International Journal of Advanced Multidisciplinary Research and Studies

ISSN: 2583-049X

Removal of Paracetamol from Aqueous Solutions Using an Eggshell Derived Adsorbent

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Abstract

Pharmaceutical residues are increasingly detected in aquatic environments, and conventional wastewater treatment does not always remove them effectively. This study evaluated an adsorbent prepared from discarded eggshells for the removal of paracetamol from aqueous solutions. Eggshells were washed, dried, ground, and thermally treated at 500 °C for 2 h. Batch experiments examined the effects of adsorbent dose (0.1–0.4 g), contact time (0–20 min), initial paracetamol concentration (10–60 mg L−1), and temperature (20–65 °C). Residual paracetamol was quantified by UV–visible spectrophotometry at 241 nm. The highest removal in the dose experiment was 85.9% at 0.2 g, whereas the greatest mass-normalized uptake reported in that series was 8.406 mg g−1 at 0.1 g. Removal reached 93.1% after 20 min and remained above 91% across the tested concentration range, with a maximum of 92.8% at 50 mg L−1. The temperature response was non-monotonic; removal ranged from 71.7% at 45 °C to 89.54% at 55 °C. The rapid initial uptake and the limited effect of additional contact time suggest that equilibrium was approached quickly under the applied conditions. Nevertheless, the non-monotonic temperature trend and inconsistencies between some reported uptake values and the stated batch volume require confirmation through replicated experiments and a complete mass-balance audit. Because eggshell is predominantly calcium carbonate, the product is described here as an eggshell-derived adsorbent rather than activated carbon unless carbon content, activation chemistry, and surface-area measurements are independently demonstrated. Overall, the material showed promising paracetamol removal, but physicochemical characterization, replication, pH-dependent testing, and validated equilibrium and kinetic modelling are needed before process-scale application.

Keywords: Paracetamol, Acetaminophen, Adsorption, Eggshell Waste, Aqueous Solution, Pharmaceutical Contaminants

1. Introduction

Pharmaceutical compounds have become an important class of emerging aquatic contaminants because their continuous consumption and incomplete removal allow parent drugs and metabolites to enter receiving waters. Early surveys established the widespread occurrence of pharmaceuticals in sewage-treatment effluents and surface waters (Ternes, 1998; Kolpin *et al*., 2002) [16, 7]. Although concentrations are often low, continuous discharge may produce chronic exposure in aquatic organisms, while the biological activity of many pharmaceuticals makes their presence environmentally relevant (Daughton & Ternes, 1999; Rivera-Utrilla *et al*., 2013) [1, 13].

Paracetamol, also known as acetaminophen, is one of the most widely used analgesic and antipyretic drugs. It can reach wastewater through human excretion, hospital discharges, manufacturing losses, and improper disposal. Biological treatment can remove a proportion of paracetamol, but performance varies with plant configuration, operating conditions, and contaminant loading. Consequently, polishing technologies such as advanced oxidation, membrane separation, and adsorption have attracted substantial attention.

Adsorption is particularly attractive because it is simple to operate, can remove dilute organic contaminants, and can be integrated into existing treatment trains. Activated carbon is the established benchmark owing to its high surface area, developed pore network, and tunable surface chemistry (Moreno-Castilla, 2004; Mansour *et al*., 2018) [11, 10]. Paracetamol uptake on carbonaceous materials may involve pore filling, dispersion forces, hydrogen bonding, and π–π or donor–acceptor

interactions. The relative contribution of these mechanisms depends on pore-size distribution, surface functional groups, solution pH, ionic strength, and the ionization state of paracetamol (Figueiredo *et al*., 1999; Nguyen *et al*., 2020) [3, 12].

Converting wastes into sorbents can reduce disposal burdens and support circular use of resources. Eggshell waste is inexpensive and abundant, but it differs fundamentally from lignocellulosic precursors: the shell consists mainly of calcium carbonate with a minor organic membrane fraction. Thermal treatment at 500 °C therefore yields a mineral-rich material rather than conventional high-carbon activated carbon unless an additional carbon source and a defined activation step are used. This distinction is important for interpreting sorption mechanisms and comparing performance with commercial activated carbon.

The present study investigated the ability of a thermally treated eggshell-derived material to remove paracetamol from water. The specific objectives were to assess the effects of adsorbent dose, contact time, initial concentration, and temperature; calculate removal efficiency and reported equilibrium uptake; and interpret the results in relation to published adsorption studies. The manuscript also identifies methodological issues that should be resolved before journal submission and scale-up.

2. Materials and Methods

2.1 Chemicals and instrumentation

Paracetamol ($C_8H_9NO_2$; molar mass 151.16 g mol−1) was used to prepare aqueous standards and test solutions. Deionized water was used throughout. Absorbance was measured with a DU 800 UV–visible spectrophotometer, and solution pH was monitored with a calibrated pH meter. An analytical balance, orbital shaker (Heidolph Promax 2020), and laboratory furnace were used during preparation and batch testing.

2.2 Preparation of the eggshell-derived adsorbent

Post-consumer eggshells were collected from households in Libya. The shells were washed repeatedly with deionized water to remove adhering material and then air-dried. The dried shells were ground and thermally treated in a furnace at 500 °C for 2 h. After cooling in a dry environment, the material was stored in a sealed container until use. Because no carbon precursor, chemical activating agent, or inert-atmosphere carbonization step was documented, the material is referred to as an eggshell-derived adsorbent.

2.3 Preparation and analysis of paracetamol solutions

A 100 mg L−1 paracetamol stock solution was prepared by dissolving an accurately weighed amount of the drug in deionized water. Working solutions were prepared by dilution. A calibration curve of absorbance against concentration was obtained at the absorption maximum of 241 nm. Blank correction and calibration linearity should be documented in the final experimental record, including the regression equation, coefficient of determination, and working range.

2.4 Batch adsorption experiments

Dose experiments were conducted using 0.1, 0.2, 0.3, and 0.4 g of adsorbent with 50 mL of paracetamol solution. The suspensions were agitated for 20 min at room temperature and the residual concentration was measured spectrophotometrically. Contact-time experiments were performed at 0, 5, 10, 15, and 20 min. Initial-concentration experiments covered 10–60 mg L−1, while temperature experiments were conducted at 20, 35, 45, 55, and 65 °C under otherwise fixed conditions. The source record did not consistently report agitation speed, pH, separation method, or number of replicates; these variables should be supplied because they materially affect reproducibility.

2.5 Calculations and data treatment

Removal efficiency and equilibrium adsorption capacity were calculated from the initial concentration C_0 and the residual concentration C_e:

$$\textbf{Removal (\%)} = [(C_0 - C_e)/C_0] \times 100$$

$$q_e = [(C_0 - C_e)V]/m$$

Where q_e is the equilibrium uptake (mg g−1), V is solution volume (L), and m is adsorbent mass (g). The reported q_e values were retained from the source dataset. However, several values do not reproduce from the stated 50 mL batch volume and therefore require verification against the laboratory notebook before submission.

Equilibrium data may be fitted to the Langmuir and Freundlich models only when sufficient independent equilibrium points are available. The linear Langmuir form is $C_e/q_e = 1/(K_l q_m) + C_e/q_m$, while the Freundlich relation is $\log q_e = \log Kf + (1/n)\log C_e$. Model selection should be based on residual analysis and error statistics rather than R^2 alone (Foo & Hameed, 2010; Tran *et al*., 2017) [4, 19]. Kinetic models should likewise be fitted to time-series data that begin with a true pre-contact measurement and include replicate uncertainty.

3. Results

3.1 Effect of adsorbent dose

Removal changed only slightly across the dose range (Table 1). It increased from 84.8% at 0.1 g to a maximum of 85.9% at 0.2 g, then decreased to 84.4% and 83.5% at 0.3 and 0.4 g, respectively. In contrast, the reported mass-normalized uptake declined from 8.406 to 2.090 mg g−1 as dose increased. Thus, 0.2 g gave the highest percentage removal, whereas 0.1 g gave the highest reported uptake per gram.

Table 1: Effect of adsorbent dose on paracetamol removal

Dose (g)	C_e (mg L−1)	Removal (%)	Reported q_e (mg g−1)
0.1	15.94	84.8	8.406
0.2	14.863	85.9	4.257
0.3	15.539	84.4	2.815
0.4	16.486	83.5	2.09

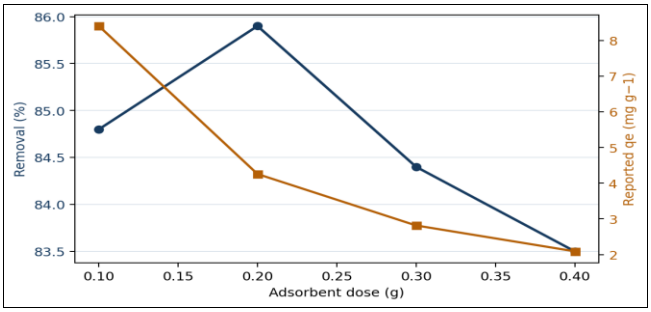

Fig 1: Effect of adsorbent dose on removal efficiency and reported uptake

3.2 Effect of contact time

Removal was already 92.8% at the first reported time point and rose marginally to 93.1% after 20 min (Table 2). The corresponding reported q_e values increased from 4.642 to 4.657 mg g−1. The narrow response indicates rapid uptake under the applied mixing conditions, but the label "0 min" should be verified because substantial removal cannot occur before adsorbent–solution contact. It may represent the first sample collected immediately after mixing rather than a genuine zero-time observation.

Table 2: Effect of contact time on paracetamol removal

Time (min)	C_e (mg L−1)	Removal (%)	Reported q_e (mg g−1)
0	7.16	92.8	4.642
5	7.16	92.8	4.642
10	7.12	92.9	4.644
15	7.01	93.0	4.65
20	6.86	93.1	4.657

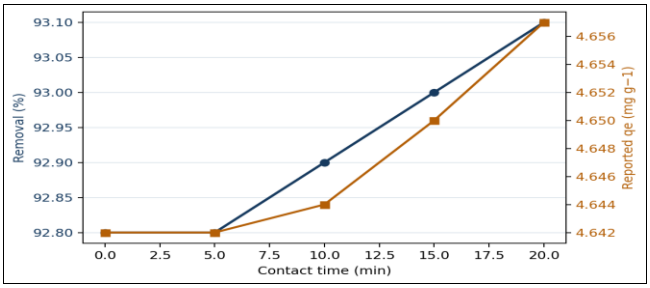

Fig 2: Effect of contact time on removal efficiency and reported uptake

3.3 Effect of initial concentration

The adsorbent maintained high percentage removal over the tested concentration range (Table 3). Removal rose gradually from 92.3% at 10 mg L−1 to 92.8% at 50 mg L−1 and then decreased to 91.7% at 60 mg L−1. The small decline at the highest concentration may indicate the beginning of site limitation. The reported uptake generally increased at concentrations above 20 mg L−1, reaching 5.274 mg g−1 at 60 mg L−1, although the 10 and 20 mg L−1 values require recalculation because they do not follow the expected mass balance.

Table 3: Effect of initial paracetamol concentration

C_0 (mg L−1)	Reported C_e	Removal (%)	Reported q_e (mg g−1)
10	7.62	92.3	2.38
20	7.61	92.4	1.239
30	7.43	92.5	2.257
40	7.27	92.7	3.273
50	7.21	92.8	4.279
60	7.26	91.7	5.274

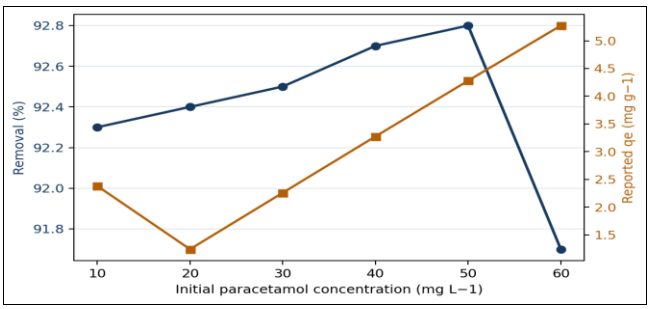

Fig 3: Effect of initial concentration on removal efficiency and reported uptake

3.4 Effect of temperature

Temperature produced a non-monotonic response (Table 4). Removal decreased from 87.6% at 20 °C to 71.7% at 45 °C, increased to 89.54% at 55 °C, and then declined to 81.7% at 65 °C. The lack of a consistent direction prevents a defensible conclusion that adsorption was simply endothermic or exothermic. Replicate measurements, temperature equilibration, and uncertainty estimates are required before calculating thermodynamic parameters.

Table 4: Effect of temperature on paracetamol removal

Temperature (°C)	C_e (mg L−1)	Removal (%)	Reported q_e (mg g−1)
20	6.18	87.6	4.69
35	7.84	84.3	4.6
45	14.13	71.7	4.29
55	5.23	89.54	4.73
65	9.15	81.7	4.54

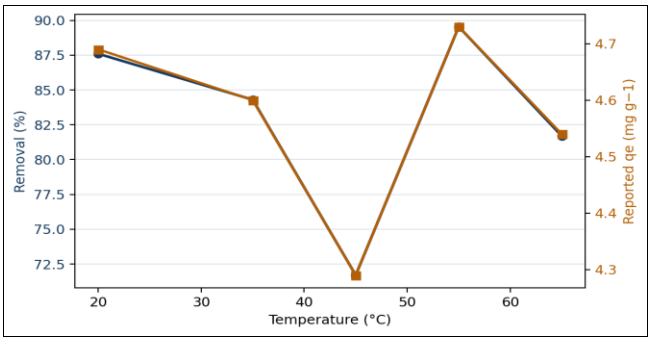

Fig 4: Effect of temperature on removal efficiency and reported uptake

4. Discussion

4.1 Influence of adsorbent dose

Increasing the amount of sorbent usually increases the total number of available sites and therefore improves percentage removal. The small rise from 0.1 to 0.2 g is consistent with this expectation. The subsequent slight decline may reflect particle aggregation, overlap of sorption surfaces, changes in suspension mixing, or normal experimental variability. Similar dose-dependent plateaus are common in batch adsorption studies because solute becomes limiting at high solid-to-liquid ratios. The simultaneous decrease in q_e with increasing dose is also expected: a larger sorbent mass shares a nearly fixed quantity of solute, leaving a greater fraction of sites unoccupied. Consequently, dose optimization should consider both treatment efficiency and uptake per unit mass rather than removal percentage alone.

Activated carbons prepared from agricultural residues have removed paracetamol effectively, but their performance depends strongly on pore structure and activation conditions. Ferreira *et al.* (2015) [2] emphasized the importance of microporosity in carbons produced from palm-derived residues, while Wong *et al.* (2018) [21] reported efficient acetaminophen uptake by carbon made from spent tea leaves. Such materials are carbon-rich and should not be compared directly with thermally treated eggshell without measuring carbon content, mineral phases, BET surface area, pore-size distribution, and surface functional groups.

4.2 Contact time and possible mass-transfer controls

The limited change after the first measurement suggests that readily accessible sites were occupied quickly. Fast early

uptake is normally driven by a high concentration gradient and abundant external sites; as adsorption proceeds, intraparticle diffusion and the decreasing number of vacant sites slow the rate. Surface heterogeneity and pore accessibility have been shown to control paracetamol adsorption kinetics on carbonaceous materials (Ruiz *et al*., 2010; Lladó *et al*., 2015) [14, 9]. However, the present time series cannot support a robust pseudo-first-order, pseudo-second-order, or intraparticle-diffusion analysis because the initial condition is ambiguous and only five closely spaced observations are available.

A good statistical fit to the pseudo-second-order equation should not be interpreted automatically as proof of chemisorption. Adsorption kinetics may involve film transfer, pore diffusion, and surface interaction simultaneously, and linearized models can distort error structure (Ho, 2006; Tan & Hameed, 2017) [6, 15]. Future experiments should include a verified zero-time concentration, early sampling within the first minutes, later observations until a stable plateau is established, and replicate measurements. A plot of q_t versus $t^{1/2}$ can then test the Weber–Morris intraparticle-diffusion model; a non-zero intercept would indicate that intraparticle diffusion is not the sole rate-limiting step (Weber & Morris, 1963) [20].

4.3 Initial concentration and equilibrium interpretation

High removal across 10–60 mg L−1 suggests that the available sorption capacity was not exhausted over most of the investigated range. Increasing initial concentration strengthens the concentration gradient at the liquid–solid interface and can raise q_e. Once active sites become limiting, percentage removal commonly declines even though uptake per gram continues to increase. The slight decline at 60 mg L−1 is compatible with this transition, but the reported C_e and q_e values must first be reconciled with C_0, V, and m.

Langmuir and Freundlich fitting is appropriate only after the mass balance has been validated. The Langmuir model assumes a finite set of energetically equivalent sites and monolayer coverage (Langmuir, 1918) [8], whereas the Freundlich equation is an empirical representation of adsorption on heterogeneous surfaces (Freundlich, 1906) [5]. Carbonaceous adsorbents frequently exhibit both energetic and structural heterogeneity, so model comparison should include non-linear regression, confidence intervals, residual plots, and error functions rather than relying solely on R^2 (Foo & Hameed, 2010; Tran *et al*., 2017) [4, 19]. No Langmuir maximum capacity or Freundlich constants are reported here because the source dataset is internally inconsistent and does not provide replicated equilibrium observations.

4.4 Temperature response and adsorption mechanism

Temperature can influence adsorption in competing ways. Higher temperature lowers water viscosity and may accelerate diffusion into pores, but it can also weaken hydrogen bonding and dispersion interactions or promote desorption. The irregular pattern observed here could reflect a combination of these effects, mineral-surface transformations, incomplete temperature equilibration, or analytical variation. Studies of acetaminophen adsorption on activated carbon have shown that temperature dependence is sensitive to surface oxidation and pore chemistry (Terzyk & Rychlicki, 2000; Terzyk, 2001) [18, 17]. A valid thermodynamic assessment requires equilibrium constants measured at several temperatures under identical conditions and converted to a dimensionless standard-state form before ΔG°, ΔH°, and ΔS° are calculated (Zhou & Zhou, 2014) [22]. The present data do not yet meet those requirements.

4.5 Nature of the eggshell-derived material

The terminology used for the sorbent is central to scientific interpretation. Conventional activated carbon is produced by carbonizing a carbon-rich precursor followed by physical or chemical activation. Eggshell mineral is predominantly $CaCO_3$, and calcination becomes substantial only as temperature and residence time increase; at 500 °C the material is expected to remain largely carbonate-rich, although the membrane and residual organics may change. Accordingly, removal may involve adsorption on mineral surfaces, hydrogen bonding with residual organic matter, acid–base interactions, or precipitation/complexation effects depending on pH. The material should be characterized by X-ray diffraction, FTIR, SEM–EDS, BET N_2 sorption, pH at the point of zero charge, ash content, and total carbon analysis. Until those measurements confirm a porous carbon phase, “eggshell-derived adsorbent” is the scientifically defensible designation.

5. Conclusions

A thermally treated eggshell-derived material removed a substantial fraction of paracetamol from the tested aqueous solutions. The best removal in the dose series was 85.9% at 0.2 g, while 0.1 g produced the highest reported uptake per unit mass. Removal reached 93.1% after 20 min and remained above 91% across initial concentrations of 10–60 mg L−1. Temperature effects were irregular, with the highest measured removal of 89.54% at 55 °C. These results indicate potential for low-cost treatment, but they do not yet establish a validated adsorption mechanism or process capacity.

Before submission to a peer-reviewed journal, the authors should verify all q_e calculations against the stated 50 mL volume, resolve the zero-time measurement, report replicate experiments with standard deviations, provide the calibration equation, document pH and agitation conditions, and characterize the solid comprehensively. Isotherm, kinetic, and thermodynamic parameters should be added only after these checks. With those additions, the study could offer useful evidence on the valorization of eggshell waste for pharmaceutical removal.

6. Declarations

Conflict of Interest: The authors declare no conflict of interest.

Funding: No funding information was provided.

Data Availability: The experimental data reported in this article are presented in Tables 1–4. Raw instrument files should be archived and made available according to the requirements of the target journal.

Author Contributions: The author-contribution statement should be completed using the CRediT taxonomy before submission.

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