

Photopolymer Storage, Reuse and Open-Vat State: Evidence Boundaries and Qualification Requirements

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Statement of Significance

A liquid photopolymer in a printer vat is not equivalent to unopened stock. It experiences a history of light, temperature, atmosphere, replenishment, mixing, build carry-out, debris and possible selective loss or redistribution of formulation components, yet elapsed time alone cannot describe that state. ER-32 defines the evidence boundaries for making a defensible reuse decision, distinguishes direct lifetime evidence from mechanistic and method evidence, and translates them into qualification gates. No unpublished 3Dresyns dataset is used to establish a numerical reuse interval.

Keywords: photopolymer storage; resin reuse; open vat; state drift; photoinitiator; inhibitor; oxygen; rheology; sedimentation; cure response; vat photopolymerisation; qualification.

Abstract

Photopolymer reuse is often governed by elapsed time or informal visual inspection, although neither variable uniquely describes the chemical and process state of a vat. This engineering review maps the composition, physical, optical, reactive and operational dimensions that can drift during sealed storage, open-vat residence and repeated production. It evaluates direct, mechanistic and method evidence, including thermal history, oxygen-inhibitor coupling, moisture exchange, suspension stability, contamination and deliberate recycling. Direct evidence for combined open-vat production ageing remains sparse, so numerical limits cannot be transferred across formulations or equipment. A four-group qualification design, sequential release gates and a claim ladder are proposed. Reuse is supported only when predefined state, print and application criteria remain equivalent to matched controls inside the declared operating space.

1. Scope and evidence architecture

ER-32 addresses the liquid or slurry state before and during vat photopolymerisation, using the process terminology of ISO/ASTM 52900 [11]. It excludes claims based only on ageing of printed parts because solid-state post-cure, residual conversion, water sorption and physical ageing do not establish liquid-resin stability. It also separates ordinary return of unused vat material from chemical recycling, mechanical recycling and deliberately recyclable resin chemistries. Those routes change formulation identity and require new material qualification [2,9].

Within the 3Dresyns Engineering Review corpus, ER-32 owns the liquid production state of a vat in service: storage, idle exposure, reuse, replenishment and carry-over. ER-30 develops oxygen inhibition, ER-36 addresses dispersion and recoating rheology of filled suspensions, ER-27 addresses conversion metrology, ER-72 addresses environmental ageing and property retention of resins and printed parts, and ER-65 addresses cationic ring-opening chemistry. ER-32 uses those mechanisms only to define the state variables and qualification logic required for reuse.

The detailed mechanism treatment is weighted towards free-radical acrylate and methacrylate chemistries, which dominate commercial vat photopolymerisation. Cationic systems based on photoacid generators, epoxides or oxetanes require separate interpretation: oxygen inhibition is generally not the governing limitation, moisture can terminate propagation, and dark reaction can continue after exposure. For cationic or hybrid formulations, elapsed time since exposure and water content may therefore be primary state variables rather than secondary covariates; see Section 3.3 and ER-65.

Evidence is classified in three tiers. Tier A consists of direct measurements on the same resin under the same vat and process history. Tier B consists of mechanistic evidence on relevant photopolymer chemistries, such as oxygen inhibition, temperature-dependent rheology or suspension stability. Tier C consists of process controls and standards that define measurement practice but do not prove resin lifetime. Only Tier A can justify a product-specific reuse interval.

Evidence tier	What it contains	Permitted use	Prohibited transfer
A - direct	Named resin, printer, vat and defined production history	Set or verify a local acceptance window	Other resins, wavelengths or vat geometries
B - mechanistic	Relevant photochemistry, rheology, thermal stability or suspension studies	Select hazards, measurements and challenge conditions	Adopt reported numerical limits as universal
C - method	Standards, artefacts and controlled measurement procedures	Define traceable tests and reporting	Treat method compliance as evidence of lifetime

2. What constitutes photopolymer state

The usable state of a vat is multidimensional. Composition includes reactive diluents, oligomers, photoinitiator, absorbers, inhibitors, pigments, fillers, dispersants and adventitious contaminants. Physical state includes temperature, viscosity, yield stress, thixotropic recovery, dissolved gases and the spatial distribution of suspended solids. Optical state includes absorbance and scattering at the printing wavelength. Reactive state includes the exposure threshold, penetration depth, polymerisation rate and maximum conversion. Operational state includes additions, filtration, cumulative illumination, printed area, peel history and residence time.

A resin can remain visually uniform while one or more of these dimensions has drifted. Conversely, a reversible viscosity change caused only by temperature need not indicate chemical degradation. Qualification therefore requires paired state and performance measurements rather than a single appearance check, as summarised in Figure 1.

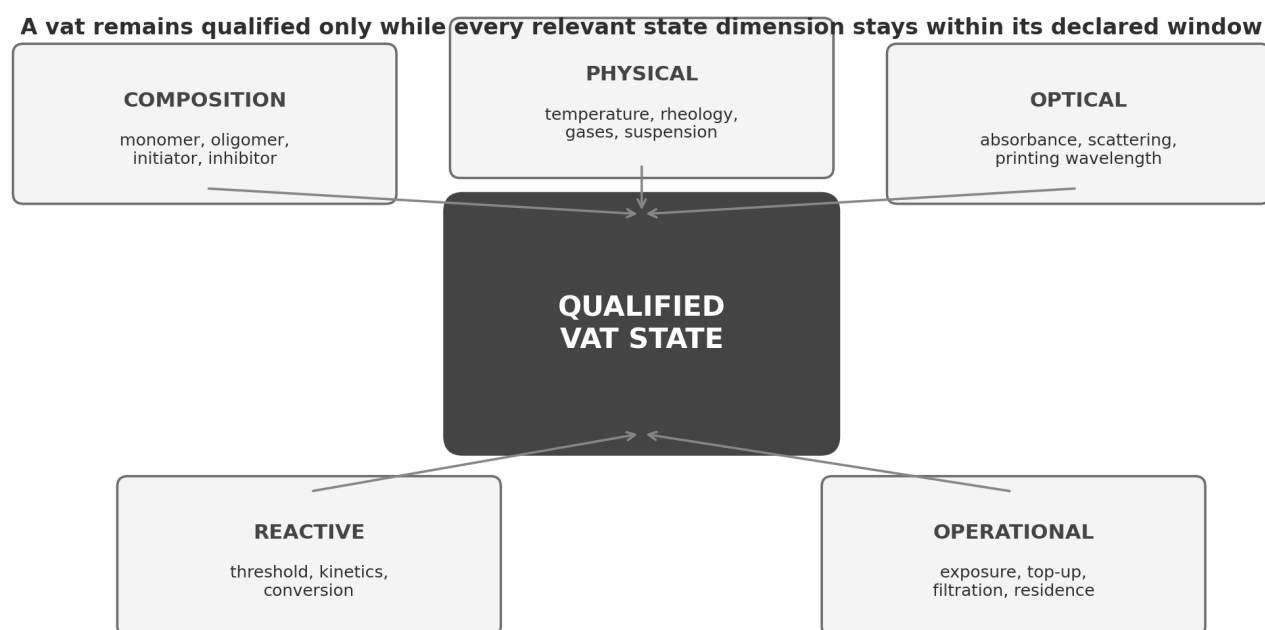


Figure 1. Five coupled dimensions define the qualified liquid state. Drift in any application-relevant dimension can trigger investigation even when appearance remains unchanged.

3. Mechanisms of state drift

The mechanisms below can act simultaneously and with different signs. Their purpose here is to identify measurements and controls, not to assign a universal degradation rate. Evidence obtained for one chemistry or process architecture remains mechanistic unless the same formulation and production history are tested directly.

3.1 Temperature and thermal history

Temperature changes viscosity, diffusion, oxygen transport and reaction kinetics. Elevated-temperature vat processing may be necessary for viscous systems, but the selected temperature must remain below conditions that promote thermally initiated reaction or volatilisation. Meenakshisundaram et al. demonstrated the value of combining rheology, differential scanning calorimetry and thermogravimetric analysis when establishing a processing window for high-viscosity vat photopolymerisation (VPP) resins [7]. Lim et al. further showed that heat-assisted VPP changes processing and printed-material outcomes for the dental resins studied [8]. Weng et al. demonstrated that process architecture can widen the usable viscosity range, confirming that a viscosity limit is inseparable from the recoating and separation system [18]. None of these studies defines a general open-vat lifetime.

Hedayati et al. observed chemical and mechanical evolution during sealed high-temperature storage of one volumetric-additive-manufacturing resin [1]. This supports thermal sensitivity as a credible state variable, while the sealed and accelerated conditions prevent direct transfer to an open production vat.

3.2 Light leakage, oxygen and inhibitor balance

Unintended exposure can consume photoinitiator, generate radicals and produce soluble oligomers, microgels or cured particles. Oxygen affects radical systems through more than one pathway. In Type II systems it can quench the excited triplet state of a sensitiser before productive co-initiation; in Type I cleavage systems its dominant effect is interception of initiating and propagating carbon-centred radicals to form less-reactive peroxy radicals. The magnitude depends on diffusion, viscosity, irradiance, photoinitiator chemistry and exposure geometry [4,5]. Oxygen-controlled dead zones can also be deliberately engineered [6]. These findings establish mechanism, not a universal open-vat lifetime.

Oxygen also participates in storage stabilisation. Phenolic inhibitors such as hydroquinone, 4-methoxyphenol (MEHQ) and hindered phenols do not efficiently intercept every carbon-centred propagating radical; an important route is interception of peroxy radicals formed after molecular oxygen adds to a carbon radical [21]. Dissolved oxygen can therefore be a co-reactant in phenolic inhibitor function rather than only an inhibitor of cure. A sealed or inerted container is not automatically the conservative state: light and contamination may be excluded while headspace and dissolved oxygen are depleted. An open vat can be re-aerated at the surface yet remain oxygen-depleted at depth, making dissolved oxygen a spatial state variable even in unfilled resin.

Inhibitor and photoinitiator concentrations are not standalone lifetime indicators. Their effective balance can change with oxygen availability, thermal history, unintended light and reactive impurities. Pereira et al. reported that storage time of uncured dimethacrylate matrices affected radical concentration in the specific formulation studied [20].

Replacement of TPO with BAPO, TPO-L, acylgermanium or other systems can also change absorption, dark stability and oxygen sensitivity. A post-TPO reformulation does not inherit the reuse behaviour of its predecessor merely because the product name is unchanged; wavelength-matched requalification is required, consistent with 3Dresyns Trilogy Paper III. A universal rule based on colour, odour or elapsed time is therefore inadequate.

3.3 Evaporation, moisture and selective composition change

An open vat can exchange matter with its surroundings. Volatile reactive diluents or solvents may be lost, while hygroscopic components may take up water. Build removal carries resin out of the vat; top-up restores bulk volume but does not necessarily restore the original ratios if components are lost selectively. Mass balance, density and viscosity trends can reveal gross changes, but chemical or spectroscopic measurements are needed when small compositional changes are consequential. A lid reduces exposure but does not erase the preceding history.

3.4 Sedimentation, creaming and agglomeration

Pigmented, ceramic, metallic and other particle-filled systems require separate control. Particle size, density contrast, surface chemistry, dispersant adsorption, yield stress and thixotropic recovery govern stability at rest and redistribution during recoating. Cramer et al. showed that dispersant selection and mixing order materially altered the rheology and suspension behaviour of silicon-nitride VPP slurries [10]. Their results support rheological and spatial sampling controls, not a universal remixing interval. A sample taken only from the surface may miss a concentration gradient near the vat floor.

3.5 Production carry-over and contamination

Printed fragments, support debris, dust, fibres, cleaning-fluid carry-over and material transferred from tools or another resin can change print reliability. Filtration removes particles above the filter cut-off but cannot restore consumed photoinitiator, lost volatiles, dissolved contaminants or altered inhibitor balance. Filter identity, nominal retention, filtered mass and visible residue should therefore be recorded. Filtration is a process step, not evidence that the resin has returned to its original state.

4. Reuse routes that must not be conflated

Route	Material operation	Evidence needed	Decision
Same-vat continuation	Material remains in one controlled vat	State trend plus reference print	Continue only within qualified window
Return to original container	Vat material is recombined with stored stock	Compatibility and contamination assessment	Do not assume the stock remains virgin
Top-up	Fresh resin added to aged vat material	Mass fractions, mixing and response after addition	Treat as a defined blend
Filtered reuse	Particles removed by declared filter	Pre/post state and filter record	Filtration cannot reset chemical age
Mechanical recycle	Cured material milled and added to liquid resin	New formulation and full property qualification	Not ordinary vat reuse [2]
Chemical closed loop	Network depolymerised or exchanged into reusable feedstock	Identity, conversion and repeated-cycle qualification	Chemistry-specific route [9]

Maines et al. showed that cured thermoset powder could be intentionally reincorporated into selected formulations and reprinted after detailed characterisation [2]. Lopez de Pariza et al. demonstrated a chemically recyclable photoprintable system based on a specific polythiourethane architecture [9]. These results are important circularity demonstrations, but they do not justify returning arbitrary cured debris or aged vat material to a conventional commercial resin.

5. Qualification study design

5.1 Required comparison groups

A defensible study should include at least: (A) unopened or freshly prepared control; (B) closed stored control from the same lot; (C) open-vat idle material exposed to the declared environment but not printed; and (D) production-aged material after a declared build history. Where relevant, separate groups should represent filtered material, top-up blends, remixed filled resin and material returned from the vat. Each group must be sampled at matched temperature and after a defined conditioning procedure.

Because Group B cannot be reconstructed after vat material is returned to the source container, draw, label and seal a retain sample at the first opening of every lot and store it under the declared reference condition. A study initiated after the source container has been opened, topped up or recombined with vat material has no valid closed stored control for that lot.

This design separates elapsed storage from open-atmosphere exposure and printing-induced carry-over, but the closed stored control is not an inert, conservative or best-case baseline. Sealed storage has its own pathway through temperature, fill fraction, headspace volume and composition, light exclusion and possible oxygen depletion. Divergence of Group B from Group A must therefore be attributed to the declared storage conditions rather than to elapsed time alone. Without Group C, a production effect cannot be separated from ambient exposure.

5.2 Controlled variables and traceability

Record resin name and lot, container identity, opening date, container fill fraction, headspace volume and headspace atmosphere, initial and remaining mass, vat identity and geometry, printer and firmware, wavelength, irradiance verification, layer thickness, build area, cumulative exposures, ambient and vat temperature, relative humidity, cover state, mixing or recoating programme, additions, filtration and abnormal events. For filled systems, define sampling depth and time after homogenisation. For inhibited radical systems, record any deliberate aeration or inerting. Do not pool histories from different vats.

5.3 Sampling discipline

Sampling itself can change the vat. Use a predefined low-shear or controlled-shear homogenisation step appropriate to the formulation, then sample at fixed locations and times. Where particle or dissolved-oxygen gradients are plausible, collect top, middle and bottom samples before and after homogenisation, including for unfilled inhibited resins. Establish method repeatability on the fresh control before ageing begins using the precision concepts of ISO 5725-1 and the basic method of ISO 5725-2 [23,24], and declare the smallest change the method can resolve. Blind the analyst to group identity for appearance and defect scoring. Store retained samples in light-controlled, labelled containers.

6. Minimum measurement package

Control block	Minimum measurement	Why it matters	Example acceptance logic
Identity and balance	Lot, history, mass in/out, additions, filtration residue	Prevents untraceable pooling and reveals gross loss	Complete traceability; unexplained loss investigated
Appearance	Colour, clarity, skin, particles, phase separation	Rapid screen for visible failure	No predefined reject feature; appearance alone never releases
Density	Temperature-controlled liquid density [13]	Supports gross composition and solids checks	Within method uncertainty and predefined control band
Rheology	Viscosity versus shear rate; yield stress or recovery where relevant [12]	Controls recoating, levelling and suspension	Equivalent process-relevant window, not one arbitrary spindle value
Chemical or optical state	Selected FTIR, UV-visible or other validated indicator	Detects conversion, absorber or composition drift	Predefined change linked to process response
Water content	Coulometric Karl Fischer titration or a validated equivalent [22]	Detects moisture uptake or loss, especially in cationic and hygroscopic systems	Predefined control band at matched sampling temperature
Stabiliser and initiator state	Inhibitor titre and residual photoinitiator by validated HPLC or spectroscopy; dissolved oxygen when meaningful	Links storage stabilisation and initiator availability to cure response	Fresh-control band interpreted with working-curve and conversion results
Cure response	Working curve or controlled thickness response at printer wavelength [3]	Detects changes in critical exposure and penetration	Dose window remains capable at declared layer thickness
Reference artefact	Same geometry, orientation and post-process [14]	Integrates resin and machine response	Dimensions, features and defects within qualified limits
Functional property	Application-critical mechanical, electrical, optical or biological endpoint	Confirms use-specific suitability	Equivalent or meets a declared specification

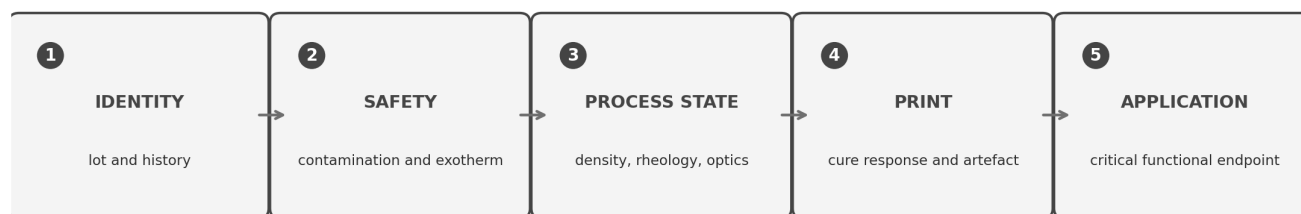
Bennett demonstrated that critical exposure, E_c , and depth of penetration, D_p , vary strongly among commercial photopolymers and with wavelength [3]. An intercomparison across 24 laboratories, each applying its own procedure to distributed material, found fitted D_p to vary by up to a factor of seven and E_c by up to a factor of seventy [19]. The aged sample must therefore be compared with its own fresh control under the same wavelength, irradiance measurement, exposure geometry and thickness method. Report the raw exposure-thickness pairs, fitting interval and residuals. Do not interpret fitted E_c or D_p alone when scattering, inhibition or departure from the logarithmic Jacobs model, $C_d = D_p \ln(E/E_c)$, makes the fit unstable [25].

7. Acceptance, quarantine and release logic

Acceptance criteria must be defined before examining the aged data. A practical hierarchy is: identity gate, safety or contamination gate, process-state gate, print-performance gate and application gate. Failure of an upstream gate prevents release even if a single printed coupon appears acceptable. Equivalence bands should reflect measurement uncertainty, process capability and the consequence of failure; statistical significance alone is not a release criterion. Table 4 lists the disposition applied at each gate, and Figure 2 summarises the sequence.

Observation	Immediate disposition	Required follow-up
Unexplained exotherm, temperature rise or abrupt viscosity increase	Immediate quarantine; do not sample or reheat	Use a safe disposal route; investigate lot, stabiliser state, temperature and headspace
Traceability incomplete or cross-contamination suspected	Quarantine	Do not merge with qualified stock; investigate or discard
Reversible temperature-related viscosity shift only	Condition and retest	Confirm density, cure response and reference print
Settling corrected by validated homogenisation	Conditional continuation	Define maximum idle time and remix procedure
Cure response or artefact drifts outside limit	Stop reuse	Investigate state, printer output and sampling; requalification required
Filter removes debris but state remains unknown	Quarantine pending tests	Filtration record plus complete state comparison
All predefined gates pass	Release within studied window	Continue trend monitoring; do not extrapolate beyond tested history

Sequential release gates



Any failed upstream gate stops release. Passing all gates supports reuse only inside the studied window.

QUARANTINE | investigate cause | recondition only under a validated procedure | otherwise dispose safely

Figure 2. Sequential release logic. A successful reference print cannot override an upstream identity, safety or process-state failure.

8. Accelerated and challenge studies

Accelerated temperature or light exposure can rank sensitivities and support mechanism selection, but acceleration factors are not valid unless the degradation pathway remains representative. Thermal analysis can identify onset regions and guide conservative test temperatures [7,15]. Laboratory light-exposure standards provide controlled source descriptions [16,17], but they address plastics generally and do not directly establish open-vat photopolymer lifetime. A challenge study should therefore include real-time production controls and verify that the same state indicators move in the same direction under accelerated and service conditions.

Avoid combining temperature, light, oxygen and contamination in the first experiment. A staged design identifies which factor drives change: single-factor screening, selected interactions, then a representative production sequence. The final claim must remain bounded by the tested resin, printer, wavelength, environment and operational history.

Accelerated thermal protocols are safety-controlled activities. Inhibited acrylate and methacrylate systems held above ambient temperature with reduced stabiliser efficacy or dissolved oxygen can undergo self-accelerating exothermic polymerisation. Set the maximum temperature conservatively against thermal-analysis onset data [7,15], limit sample mass, use suitable secondary containment and temperature monitoring, and do not run tests unattended. Do not inert the headspace of an inhibited radical formulation unless the supplier has explicitly validated that condition. Treat any unexplained exotherm, temperature rise or abrupt viscosity increase as an immediate quarantine event, not as an ordinary data point.

9. Claim levels and minimum reporting dataset

A result should be expressed at the lowest claim level supported by the evidence. Passing one print after storage is an observation, not a validated lifetime. Repeated agreement with fresh controls under a declared protocol can support a bounded reuse window. A transferable public claim requires evidence across the declared sources of variation, including lots, printers or vats when those factors are inside the claim.

Claim level	Minimum evidence	Permitted wording	Boundary
Observation	One documented aged sample and matched control	The tested sample printed under the stated conditions	No lifetime or reuse-count claim
Controlled comparison	Replicates, matched histories and predefined measurements	No detected material difference within the stated method	Applies only to the tested time point
Qualified local window	Time series, acceptance gates and process challenge in one defined system	Reuse accepted up to the tested boundary	Named resin, lot strategy, printer, vat and environment
Transferable product claim	Multiple lots and representative equipment, environments and operators	Product instruction within the validated operating space	No extrapolation outside the declared space

Table 1 and this claim ladder are complementary. Table 1 prohibits extrapolation of Tier A results to configurations never tested, whereas a product-level claim is permitted only inside an operating space whose relevant variation in lots, printers, vats, environments and operators was sampled. A transferable claim inside a validated space is not permission to transfer a result outside that space.

Every study report should provide the complete material identity and lot, control definitions, vat and printer identifiers, wavelength and measured irradiance, environmental history, sampling and conditioning procedure, additions and removals, raw replicate results, calculation method, uncertainty or repeatability estimate, deviations, failed builds and the exact boundary of the conclusion. Reporting only averages, successful coupons or fitted working-curve parameters prevents independent assessment. Photographs and spectra should retain scale, acquisition conditions and sample identity.

A result of 'no significant difference' is not automatically evidence of equivalence. The study must have sufficient precision and replication to exclude changes larger than the predefined practical limit. Equivalence may be assessed using two one-sided tests (TOST) against a predeclared equivalence margin, delta, fixed in the protocol and justified by process capability and consequence of failure [26]. Conversely, a statistically detectable change can remain operationally acceptable if it lies inside the justified equivalence band and all downstream gates pass. Criteria and analysis must be fixed before reviewing the outcome.

10. Research priorities

The highest-value studies are factorial open-vat time series that separate idle exposure from printing history; quantify mass and compositional balance during repeated builds and top-up; map top-to-bottom concentration in filled systems; relate inhibitor or photoinitiator indicators to Ec, Dp and defect yield; compare filtration before and after controlled debris challenges; and test whether accelerated protocols predict real production drift. Public reporting should include negative results and measurement uncertainty so that apparent stability is not confused with inadequate sensitivity.

11. Conclusions

Photopolymer reuse is a state-equivalence decision, not a calendar rule. Sealed storage, elevated temperature, oxygen-inhibitor coupling, moisture exchange, suspension rheology and deliberate recycling are supported by published evidence, but none supplies a universal open-vat lifetime. The combined production state must be measured against a fresh control and a closed stored control while recognising that neither is inert and each carries its own ageing pathway. Appearance and filtration are useful screens but cannot release material alone. A defensible reuse claim requires traceable history, predefined equivalence margins, matched sampling, cure-response testing, a reference print and every application-critical endpoint. Results apply only within the studied resin, printer, vat, wavelength, environment and operational window.

Appendix A. Implementation in a commercial materials programme

For 3Dresyns materials, reuse instructions should be linked to the exact product technical data sheet (TDS), instructions for use (IFU) revision and printing system. A generic statement such as 'filter and reuse indefinitely' is not supportable. Product-specific documentation may define permitted conditioning, mixing, filtration, maximum interruption, top-up policy and verification artefact only when those controls have been validated. Highly filled, moisture-sensitive, volatile, biologically qualified or composition-critical materials require narrower controls than a simple unfilled model resin.

The minimum commercial record is a vat-history identifier linked to resin lot, printer, dates, build count or cumulative build area, additions, filtration, storage state and verification result. Material with unknown history should not be pooled into controlled stock. Customer observations can trigger investigation but do not replace controlled evidence.

Limitations

This is a qualitative engineering review, not a systematic meta-analysis. Direct peer-reviewed studies of commercial liquid resins under repeated open-vat production histories remain scarce. Several cited studies provide mechanistic or method evidence rather than direct lifetime data. No unpublished 3Dresyns dataset is used to establish a numerical reuse interval.

Declarations

Funding: none declared. Competing interest: the author is Founder and Scientific Director of 3Dresyns by Resyner Technologies S.L. Commercial implementation examples are separated from independent evidence. No new experimental dataset was generated. All claims, citations, standard editions and evidence boundaries were verified by the author, who takes full responsibility for the content and release approval.

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